NIST SPECIAL PUBLICATION **260-134**

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Standard Reference Materials®

Standard Reference Material 1749: Au/Pt Thermocouple Thermometer

Dean C. Ripple and George W. Burns

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Issued March 2002

National Institute of Standards and Technology Special Publication 260-134
Natl. Inst. Stand. Technol. Spec. Publ. 260-134, 42 pages (March 2002)
CODEN: NSPUE2

U.S. GOVERNMENT PRINTING OFFICE
WASHINGTON: 2002

For sale by the Superintendent of Documents, U.S. Government Printing Office
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Abstract

Standard Reference Material® (SRM®) 1749 is a lot of 18 specially-constructed gold versus platinum (Au/Pt) thermocouple thermometers, each calibrated on the International Temperature Scale of 1990 (ITS-90) and supplied with integral lead wires and a protective sheath. It is the most accurate thermocouple available over the range 0 °C to 1000 °C, with an expanded uncertainty (k=2) less than 8.3 m°C from 0 °C to 962 °C, and rising to 14 m°C at 1000 °C. We describe the fabrication and calibration methods used to prepare the SRM 1749 thermocouples. These thermocouples are exceptionally thermoelectrically homogeneous and stable over hundreds of hours of use, making them an excellent choice as a secondary reference thermometer. To facilitate low measurement uncertainties in applications, practical methods for emf measurements and comparison calibrations of other thermometers with the SRM 1749 thermocouple are also given.

Disclaimer

Certain commercial equipment, instruments or materials are identified in this paper in order to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by NIST, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Acknowledgment

The authors wish to thank the Standard Reference Materials Program for their support in the development of this new SRM.

Table of Contents

1. Introduction 1
2. Properties of Au/Pt thermocouples 1
3. Fabrication of the thermocouples 2
4. Calibration of the thermocouples 4
5. Uncertainty of the thermocouple calibration 6
   5.1 Thermocouple reproducibility 7
   5.2 Emf measurement uncertainties 8
   5.3 Realization of the ITS-90 11
   5.4 Thermocouple inhomogeneity 11
   5.5 Uncertainty of the reference function 12
   5.6 Uncertainty of the ice point 13
   5.7 Summary of uncertainties 13
6. Properties of thermocouples prepared for SRM 1749 13
7. Mathematical methods for use with SRM 1749 17
8. Maintenance of the thermocouple 19
9. Preparation of an ice bath for the reference junctions. 19
10. Emf measurements 20
    10.1 Digital voltmeters 20
    10.2 Scanners 21
    10.3 Junction boxes and wiring 22
    10.4 Methods for data acquisition 23
11. Preventing thermocouple contamination 24
12. Comparison measurements with the Au/Pt thermocouple 24
13. References 25

Appendix: Certificate for SRM 1749 27

List of Tables

Table Number | Page

1. Calibration uncertainties for the SRM 1749 thermocouples 7
2. Subcomponents of the gain uncertainty for the digital voltmeter 10
3. Sample coefficients, a_i, of a calibration function for SRM 1749 thermocouples 17
4. Temperature-emf data pairs evaluated from the coefficients in Table 3 18
5. Coefficients c_i of an approximate inverse function for Au/Pt thermocouples 18

List of Figures

Figure Number | Page

1. Photographs of a SRM 1749 thermocouple 3
2. Thermal expansion coil at the measuring junction of a Au/Pt thermocouple 4
3. The difference between the measured emf values for a typical SRM 1749 thermocouple and the reference function for Au/Pt thermocouples 5
4. Calibration uncertainties for the SRM 1749 thermocouples 7
5. Non-linearity of a digital voltmeter at low voltage values 8
6. Non-linearity of a digital voltmeter on the range with full scale voltage of 1.2 V 9
7. Fractional deviation of measured voltage from value of voltage expected by scaling, as a function of temperature change 10
8. Values of emf on insertion into and withdrawal from the aluminum freezing-point cell during a freeze 11
9. Values of emf on insertion into and withdrawal from the silver freezing-point cell during a freeze 12
10. Differences between the measured emf values for the SRM 1749 thermocouples and the reference function 14
11. Correlation of emf values at the aluminum point, E(Al), with emf values at the silver point, E(Ag) 14
12. Correlation of emf values at the ice point, E(Ice), with emf values at the indium point, E(In) 15
13. Ratio of the standard deviation of emf values in the lot of SRM 1749 thermocouples to the standard deviation for the reproducibility with thermal cycling 15
14. Emf value at the silver freezing point of a thermocouple fabricated at the same time and from the same wire lots as the SRM 1749 thermocouples, and used as a check standard 16
15. Inhomogeneity and change in the emf measured at the silver freezing point for an SRM 1749 thermocouple that was heated at 980 °C 17
16. Schematic cross section of a junction box with low thermal emfs, constructed with commercial binding posts 23

1. Introduction

The Standard Reference Material® (SRM®) 1749 thermocouple is a specially-constructed and annealed gold versus platinum (Au/Pt) thermocouple, with integral lead wires and a protective silica glass sheath. It is the most accurate thermocouple available over the range 0 °C to 1000 °C, with an expanded uncertainty (k=2) less than 8.3 m°C from 0 °C to 962 °C, and rising to 14 m°C at 1000 °C. In this range, other common thermometer types are platinum-rhodium alloy thermocouples, and platinum resistance thermometers. Relative to platinum-rhodium alloy thermocouples, the SRM 1749 thermocouple is over an order of magnitude more accurate and more homogenous, at the cost of being more expensive and somewhat more difficult to use. Relative to Standard Platinum Resistance Thermometers (SPRTs), the defining instrument of the International Temperature Scale of 1990 (ITS-90) [1] from 13.6 K to 962 °C, the SRM 1749 is substantially more rugged but slightly less accurate. Relative to platinum resistance thermometers with a more rugged construction than SPRTs, the SRM 1749 thermocouple has similar calibration uncertainties, is somewhat more resistant to loss of calibration, and has a higher upper temperature limit.

The combination of ruggedness, a calibration uncertainty significantly less than almost all industrial thermometers, and long-term stability of its calibration make the SRM 1749 thermocouple ideal for use as a secondary reference standard.

This document serves two purposes: to provide guidance in the proper use and care of the SRM 1749 thermocouple thermometer and to document the methods used at NIST for its fabrication and calibration. Section 2 gives background on the general properties of Au/Pt thermocouples; Sections 3 through 5 describe the fabrication, calibration, and uncertainty of the SRM 1749 thermocouples in particular, and Sections 6 through 12 give instructions on the care and proper use of the SRM 1749 thermocouple.

2. Properties of Au/Pt thermocouples

Although the SRM 1749 thermocouple operates on the same physical principles as all other thermocouples, advances in the fabrication, annealing, and calibration techniques result in a thermocouple thermometer that has vastly superior accuracy and homogeneity relative to other thermocouples over the range 0 °C to 1000 °C.

It is informative to compare the SRM 1749 with more traditional thermocouple designs. Thermocouples constructed from platinum-rhodium alloys and pure platinum are currently the predominant choice for use as a secondary reference standard. Type S (Pt-10%Rh vs. Pt) and type R (Pt-13%Rh vs. Pt) thermocouples cover the temperature range from 0 °C to approximately 1400 °C, and type B (Pt-30%Rh vs. Pt-6%Rh) thermocouples generally are used over the range from 800 °C to 1700 °C [2]. Compared to thermocouples manufactured from "base" metals, such as the type K thermocouple, the platinum-rhodium alloy thermocouples have superior stability and initial calibration accuracy. However, the highest accuracy obtainable with any of the platinum-rhodium alloy thermocouples is 0.1 °C to 0.2 °C at 1100 °C as a consequence of preferential oxidation of rhodium [3-6]. Rhodium will form a stable oxide within a temperature band from approximately 550 °C to 900 °C. As the rhodium oxide forms, a thermoelement formed from a platinum-rhodium alloy will become depleted in rhodium, and changes in thermoelement composition will result in changes of the emf-temperature relationship and in thermoelectric inhomogeneity of the thermoelement.

Thermocouples constructed from pure elements, the Au/Pt or Pt/Pd thermocouple for example [7,8], do not suffer from preferential oxidation problems. This fact has several important consequences: pure element thermocouples are inherently more thermoelectrically homogeneous, and their thermoelectric stability is not limited by shifts in alloy composition caused by preferential oxidation. Additionally, because pure element thermocouples do not require adjustments of alloy composition to match a reference function, the interchangeability of thermocouples manufactured from sufficiently pure elements is excellent and the deviations of actual thermocouples from the appropriate reference function are small. In the case of Au/Pt thermocouples, homogeneity and initial calibration tolerances are superior to those of a type S or type R thermocouple by over an order of magnitude, and no long-term drift has been detected over 1000 h of use [7].

Pure element thermocouples do have some limitations. Special construction techniques are necessary to minimize mechanical strain caused by the different thermal expansion coefficients of the pure element thermoelements. Because the pure elements used have lower melting points than platinum-rhodium alloys, the upper limit of use of the thermocouples is 1000 °C for Au/Pt thermocouples and 1500 °C for Pt/Pd thermocouples. In certain applications, the high thermal conductivity of a gold thermoelement may be a disadvantage.

3. Fabrication of the thermocouples

The SRM 1749 thermocouples were constructed from 0.5 mm diameter gold and platinum wire of the highest purity available, typically 99.999 % mass fraction. The high purity of the platinum wire was attested to by its close thermoelectric agreement with the NIST-maintained platinum thermoelectric standard, Standard Reference Material SRM-1967, commonly referred to as Pt-67 [9]. The emf of the platinum wire versus Pt-67 at 1064 °C, with the reference junction at 0 °C, was found to be +0.6 μV. The fabrication techniques followed previous practice [8,10].

The annealing of the gold and platinum wire is a critical process in producing thermoelements that are thermoelectrically stable and uniform along the length of the thermoelements. High temperature annealing serves two purposes: First, the annealing relieves any mechanical strains in the wire. Second, annealing may reduce the quantity of impurities in the wire by either driving off volatile impurities or by oxidizing reactive impurities. Two common methods of annealing are either to anneal the wire electrically by suspending a loop of wire from two clips and heating the wire by passing AC current through the wire [11], or to anneal the wire by placing the wire in a uniform zone of a furnace. Gold has insufficient mechanical strength to reliably anneal it by an electrical anneal, so it was annealed only in a furnace. Platinum was annealed by both methods. Prior to furnace annealing, the platinum wires were electrically annealed in air for 10 h at 1300 °C and 1 h at 450 °C. The wires were furnace annealed by pulling segments of the wire into the bores of a high-purity (99.8 % mass fraction) alumina insulator tube. A separate alumina tube is dedicated for use with each metal. The alumina tube holding the wires was placed into a larger diameter alumina protecting tube mounted in a 1.1 m long horizontal tube furnace. The wire segment length was short enough that during a furnace anneal the wire was located in a zone of the furnace that was uniform in temperature to within 2 °C, ensuring that the wire will be uniformly annealed over the full length. Furnace annealing schedules were 10 h at 1000 °C and overnight at 450 °C for gold, and 1 h at 1100 °C and overnight at 450 °C for platinum.

Following annealing, pull wires were used to thread the thermoelements into the 1.6 mm bores of a twin bore, high-purity alumina tube with overall diameter of 4.7 mm and length of 76 cm. A relatively long tube length is useful for the maintenance procedures described in Section 8 below. A relatively large bore diameter allows the thermoelements to move easily through the bores as they expand with heating. Before use, all alumina tubes were baked for 50 h in air at 1100 °C. During assembly of the thermocouple, the thermoelement segments were rejoined by butt-welding with a small hydrogen-oxygen torch. As shown in Fig. 1, the thermocouple wires emerging from the alumina tube were insulated with flexible fiber-glass tubing to within 1 cm of their ends, and the fiber-glass tubing was joined to the alumina tube with heat-shrinkable sleeving. A cylindrical sleeve of soft copper was crimped over the fiber-glass tubing where the thermoelements emerged from the insulator. This crimp tube compresses the fiber-glass tubing against the thermoelements to anchor them near the end of the alumina tube. For each thermocouple, a four or five turn coil of 1 mm diameter constructed from 0.12 mm diameter platinum wire was used to connect thermoelements at the measuring junction, as seen in Fig. 2. A pair of insulated copper wires was soldered to the other ends of the thermoelements to form the reference junctions. Both copper wires were cut from one spool to minimize any thermal emf caused by slight mismatches in the copper composition. The positive lead of the thermocouple, connected to the gold wire, is color coded with a small piece of yellow insulation.

The portion of the alumina-sheathed section of the thermocouple is further protected by a silica glass sheath of 7 mm outer diameter, as shown in Fig. 1. Sufficient room is left at the end of the sheath to allow free expansion of the thermoelements on heating to 1000 °C. The silica glass sheath may be removed by loosening the O-ring fitting at its open end, but the coil of the SRM 1749 thermocouple is extremely vulnerable to damage in this state. Only highly trained operators should use the SRM 1749 thermocouple without its outer sheath. The flexible portion of the thermocouple is further protected by an outer sheath of fiberglass sleeving both over the gold and platinum thermoelements and over the copper leads.

The reference junctions of the thermocouple are mounted in a single stainless steel tube, closed at one end. The recommended method for use of this reference-junction assembly is discussed in Section 9. This reference-junction assembly should never be disassembled, since disassembly and reassembly risks mechanically straining the wires.

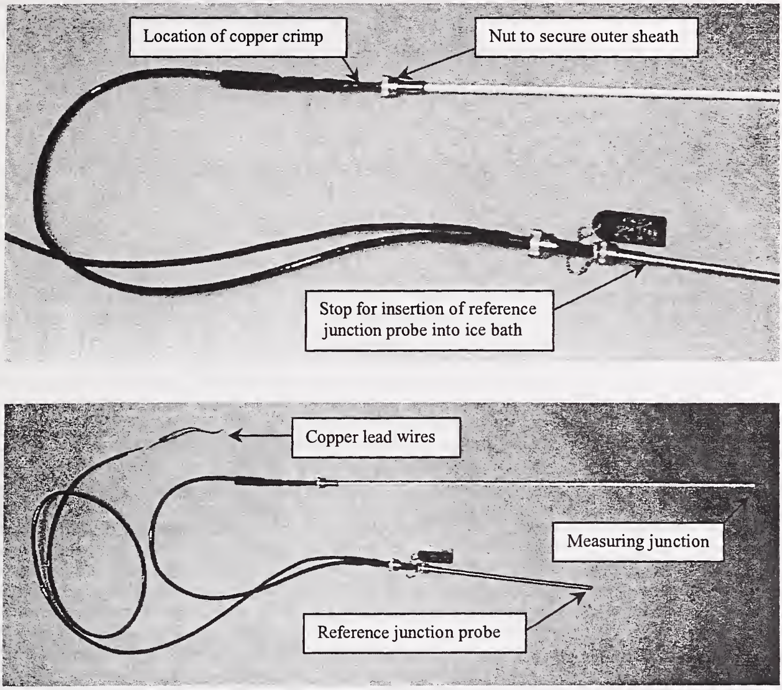

Figure 1. Photographs of a SRM 1749 thermocouple, with key components identified.

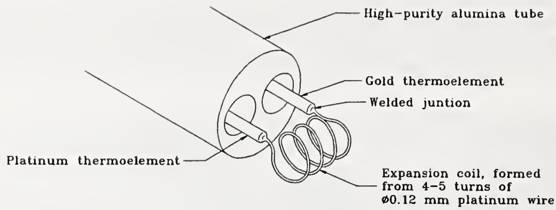

Figure 2. Schematic drawing of the thermal expansion coil at the measuring junction of a Au/Pt thermocouple.

4. Calibration of the thermocouples

After construction but prior to full calibration, each thermocouple was furnace annealed in air for 1 h at 1000 °C, cooled to 450 °C over a period of approximately three hours, and held at 450 °C for at least 10 h. Then, the effects of inhomogeneity of the thermocouple were determined by measuring its emf on insertion into and withdrawal from a silver freezing-point cell during a freeze of the silver. For this measurement, the test thermocouple was inserted into the cell, after a freeze was induced, to a point where the measuring junction was held 2 cm above the surface of the silver. After 25 min at this position, the thermocouple was inserted in steps of 2 cm every 5 min. The thermocouple was held at full immersion (18 cm below the surface of the silver) for 25 min, and then withdrawn in steps of 2 cm every 5 min, after an initial step of 1 cm. At immersions greater than 8 cm, the thermocouple measuring junction was in thermal equilibrium with the freezing metal. The temperature of the freezing metal varies slightly with the depth of immersion, as a consequence of the hydrostatic head, but variations in the emf from this effect are less than the equivalent of 1 m°C for immersions ranging from 8 cm to 18 cm. Measurements of very similar freezing point cells with platinum resistance thermometers clearly indicate that for sufficient immersion into the cell, the temperature varies only slightly as the thermometer immersion is varied, as predicted by the variation in the hydrostatic head. The signal generated by a thermocouple, however, depends not only on the temperature of the measuring junction, but also on the thermoelectric properties of the thermoelements. The emf is generated in regions where the thermoelements pass through thermal gradients. If, as a consequence of chemical or physical variations, the thermoelectric properties vary along the length of the thermoelement, the thermocouple is said to be "inhomogeneous." The emf generated by an inhomogeneous thermocouple will vary with depth of immersion into a fixed-point cell because different sections of the thermoelements will be exposed to the regions of thermal gradients in the furnace.

Thus, in the measured immersion curves into the aluminum or silver fixed-point cell, any deviation from a constant value of emf was taken as an indication of inhomogeneity or drift of the thermocouple (short term drift in the emf measurements is negligible). The spread in emf values for all measurements at immersions of 8 cm and greater was calculated and defined as the inhomogeneity. All of the SRM 1749 thermocouples had inhomogeneity values at the silver freezing point (961.78 °C) less than 0.17 μV, equivalent to 7 m°C, and hysteresis values less than –0.14 μV, equivalent to 6 m°C. These values are extreme limits; the rms value for inhomogeneity for the SRM 1749 thermocouples is 0.050 μV at the silver freezing point, equivalent to 2 m°C.

A reference function for Au/Pt thermocouples on the ITS-90 has been measured at NIST over the range 0 °C to 1000 °C [7]. The emf versus temperature responses of the SRM 1749 thermocouples deviate from the reference function by no more than 16 m°C. To attain the minimum possible uncertainty with this thermometer, each individual SRM was calibrated, and a unique calibration function was generated.

Au/Pt thermocouples can readily be calibrated by measuring the thermocouple emf at fixed points defined on the ITS-90. At NIST, the fixed points used are ice (0 °C), indium (156.5985 °C), tin (231.928 °C), zinc (419.527 °C),

aluminum (660.323 °C), and silver (961.78 °C). Each of the fixed-point cells used had previously been compared against the corresponding reference cell maintained in the NIST Resistance Thermometer Calibration Laboratory. At the fixed points of aluminum and silver, a full immersion curve was obtained on each thermocouple to document the inhomogeneity of the thermocouple. The measured emf values at full immersion into each of the fixed point cells were used to determine the calibration function for each thermocouple. Results for a typical thermocouple are shown in Fig. 3. Emf values at less than full immersions were used to determine the uncertainty component for thermocouple inhomogeneity. Between each fixed-point measurement, the SRM 1749 thermocouples were annealed at 450 °C overnight, as described in Section 8.

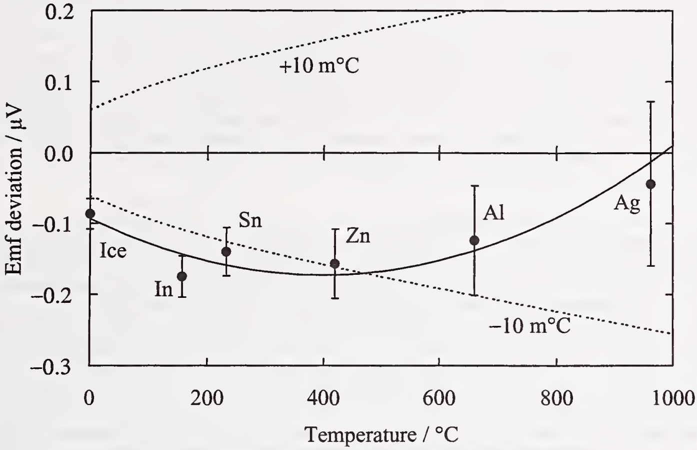

Figure 3. The difference between the measured emf values for a typical SRM 1749 thermocouple and the reference function for Au/Pt thermocouples, defined as the emf deviation, is plotted versus temperature. Solid circles indicate measured data, the solid line is a quadratic fit to the data, and the error bars indicate one standard uncertainty at each fixed point. The dotted lines show the emf deviation equivalent to ±10 m°C.

The cell design and the design of the furnaces used are discussed in Ref. [11] and [12]. The well of each freezing-point cell contained a silica glass protection tube (8 mm outer diameter, 6 mm inner diameter) closed at one end, open to air at the other end, and matte finished on the outer surface to minimize heat losses by radiation piping. At full immersion of the thermocouple into the freezing-point cells, the measuring junction of the thermocouple was 18 cm below the surface of the metal. Freezes in the gold, silver, and aluminum cells were performed in a vertical furnace containing a 61 cm long sodium heat pipe. Freezes in the zinc, tin, and indium cells were performed in a single zone furnace having an aluminum moderating block. For all of the freezing points, the furnace was held 0.5 °C below the freezing-point temperature after the freeze was initiated, and the freeze was induced internally by inserting an alumina insulator, initially at room temperature, into the cell.

Measurements of the thermocouples at the ice point used a properly prepared ice bath (see Section 9 of this document) contained in a cylindrical Dewar flask (7 cm inner diameter and 41 cm deep). The test thermocouple was contained in a closed-end glass tube (7 mm outer diameter, 5 mm inner diameter) inserted in the tightly packed mixture of ice and water in the flask. Its measuring junction was located 35 cm below the surface of the ice-water mixture when measurements were made.

All of the emf measurements were made with a calibrated digital multimeter (Hewlett-Packard model 3458A) and scanner (Hewlett-Packard model 3495A) system. The thermocouple data were obtained automatically via a computer-controlled IEEE-488 bus and logged to a data file for later analysis. The reference junctions of the thermocouples were maintained at 0 EC in an ice bath when measurements were made.

To obtain a mathematical calibration function of each thermocouple, emf values computed from the reference function were subtracted from the emf values measured at each fixed point. The resulting emf deviation was then modeled by a quadratic function of temperature. Coefficients of the quadratic function were determined by the method of least squares, and addition of these coefficients to those of the reference function gave the calibration function for the particular thermocouple under test. For the least squares fitting procedure, the calibration points were weighted by the inverse square of the reproducibility of the thermocouples, measured as the standard deviation of a set of measurements at each fixed point. The reproducibility values used had been obtained from a previous study of the reproducibility of Au/Pt thermocouples at fixed points [7]. The results of the least squares fitting of the quadratic model to the data gave values of the reduced chi-squared statistic of 0.60±0.37, which is somewhat less than the expected value of one. We believe that this is primarily because the reproducibilities had been evaluated for data including multiple thermal cycles over a period of many months, but the SRM 1749 data included thermal excursions to only one round of fixed-point temperatures over a briefer period of time. It is also possible that our proficiency in fabricating, annealing, and measuring Au/Pt thermocouples has improved since the original measurement of reproducibilities at fixed points.

With most thermocouples whose reference junctions are maintained at 0 °C, calibration at the ice point is not common practice—the emf value at this point is often simply assumed to equal zero. In fact, all thermocouples will have a non-zero emf value at the ice point as a result of small inhomogeneities in the thermoelements or copper lead-wire from which the thermocouple thermometer has been constructed. For thermocouples that have no connections or discontinuities of the thermoelements, this effect is small, and measurement of the ice point as part of the calibration is generally necessary only for pure element thermocouples of the highest level of accuracy. In the case of SRM 1749, there are three main sources of wire inhomogeneity. First, the copper lead wires from the ice point to the emf-measuring apparatus are neither as well annealed nor as chemically pure as the gold and platinum thermoelements. Minor differences between the two leads will result in a small emf contribution when the lead wires pass from room temperature to 0 °C. Second, as a result of the annealing process for the gold and platinum wires, there may be slight differences in the thermoelectric properties of the thermoelements at the reference junction end of the thermocouple relative to the properties at the measuring junction end of the thermocouple. Third, all of the wires at the reference junctions are subject to slight mechanical strain when the reference-junction assembly is fabricated.

5. Uncertainty of the thermocouple calibration

For a thermocouple of ideal properties, the process of calibration requires the measurement of the thermocouple emf while simultaneously maintaining the reference and measuring junctions at known temperatures. The data pairs of temperature versus emf could then be fit by a polynomial equation that would give a temperature-emf relation. The uncertainty of this relation at the time of test would depend only on the uncertainty of the emf measurement and the uncertainties related to the determination of the thermocouple junction temperatures.

What is of more interest to the user is knowledge of the uncertainty of the temperature-emf relation when the thermocouple is used in a different apparatus at a later date. For this, it is necessary to add components for thermocouple reproducibility and inhomogeneity to the uncertainty budget. We have chosen to construct the uncertainty budget to include these components, defining the inhomogeneity component so that the combined uncertainty can be thought of as the uncertainty of the temperature-emf relation when used in a measurement environment similar, but not identical, to the NIST measurement system. Cases for which the user environment can no longer be considered similar to the NIST system are discussed below, in sections 5.4 and 5.6.

Table 1 lists the calibration uncertainty of the SRM 1749 thermocouples at fixed point temperatures and a number of additional temperatures. Each of the subcomponents of uncertainty mentioned in Table 1 are discussed below, and

the dominant components are shown in Fig. 4. The uncertainties have been evaluated in accordance with Ref. [13]. Except for the component labeled "Thermocouple reproducibility," all of the components are Type B uncertainties.

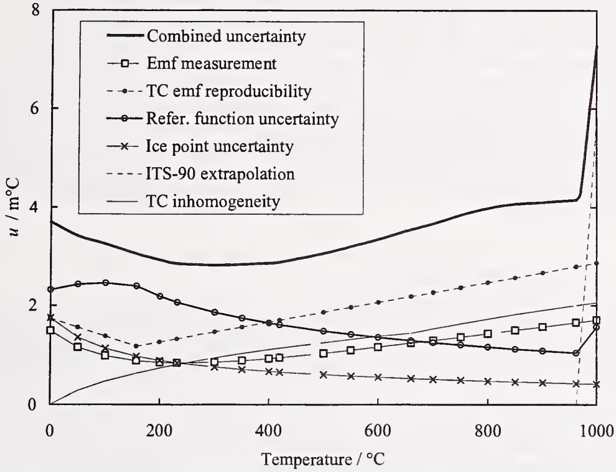

Figure 4. Calibration standard uncertainties and subcomponent uncertainties for the SRM 1749 thermocouples. Only subcomponents that are significant over some range of temperature are shown.

Table 1. Calibration uncertainties for the SRM 1749 thermocouples.

	All uncertainties expressed in millidegrees Celsius								
t /°C	Emf measur.	TC reproduc.	Realization of ITS-90	Uncertainty of reference function	ITS-90 non-unique. & extrapolat.	Ice point uncertainty & inhomog.	TC inhomog.	Combined std. uncert. *u*	Expanded uncertainty *U*(*k*=2)
0	1.49	1.7	0.10	2.3	0.00	1.75	0.00	3.7	7.4
100	0.98	1.4	0.25	2.5	0.29	1.13	0.47	3.3	6.5
156.599	0.89	1.2	0.25	2.4	0.00	0.97	0.63	3.1	6.1
200	0.85	1.3	0.25	2.2	0.29	0.89	0.74	2.9	5.9
231.929	0.84	1.3	0.24	2.1	0.00	0.84	0.81	2.9	5.7
300	0.85	1.5	0.31	1.9	0.29	0.76	0.94	2.8	5.6
400	0.93	1.7	0.42	1.6	0.14	0.67	1.11	2.9	5.7
419.527	0.95	1.7	0.44	1.6	0.00	0.66	1.14	2.9	5.8
500	1.04	1.9	0.79	1.5	0.28	0.60	1.25	3.1	6.1
600	1.16	2.1	1.23	1.4	0.20	0.55	1.38	3.3	6.7
660.323	1.24	2.2	1.50	1.3	0.00	0.53	1.44	3.5	7.1
700	1.29	2.3	1.46	1.3	0.60	0.51	1.53	3.7	7.3
800	1.43	2.5	1.36	1.2	1.15	0.47	1.73	4.0	8.0
900	1.57	2.7	1.26	1.1	0.81	0.44	1.91	4.1	8.2
961.78	1.66	2.8	1.20	1.0	0.00	0.42	2.00	4.2	8.3
1000	1.71	2.9	1.16	1.6	5.77	0.41	2.06	7.3	14.5

5.1 Thermocouple reproducibility

The reproducibility of emf measurements in fixed-point cells of a set of Au/Pt thermocouples, with construction very similar to the SRM 1749 thermocouples, was determined in the original work on the reference function for Au/Pt thermocouples [7]. This reproducibility was taken as the Type A uncertainty for the calibration of the SRM 1749 thermocouples. When expressed as an equivalent temperature uncertainty, the Type A uncertainty could be represented by the expression $u_A = 0.201\times10^{-5}(t\ /°C) + 0.86$ m°C for the fixed points indium, tin, cadmium, zinc, aluminum, and silver. The Type A uncertainty for the ice point was determined from repeat measurements on several thermocouples in the SRM 1749 lot. The value found was $u_A(\text{ice}) = 1.74$ m°C. In Table 1, this uncertainty is termed the "Reproducibility of Au/Pt TCs", but more accurately can be considered to include also effects of the reproducibility of the emf measurements, the reproducibility of the reference junction bath, the thermoelectric instability of the thermocouples over the course of the calibration measurements, and the reproducibility of the fixed-point realizations.

5.2 Emf measurement uncertainties

Uncertainties of the emf measurements not covered in the Type A uncertainty were determined by independent measurements of the variation of the thermal emfs from the scanner relays and wiring, measurement of the voltmeter non-linearity, measurements of the gain stability of the multimeter over extended periods of time, and by intercomparison of the multimeter with other multimeters of the same and different manufacturer.

Figures 5 and 6 display typical results for the voltmeter nonlinearity, as measured on a Resistance Bridge Calibrator (RBC) [14]. The RBC is similar to a Hamon network of resistors [15], but is configured in such a way as to allow up to 35 independent resistance values to be obtained from serial and parallel combinations of only four resistors. Because of this large redundancy of resistance values, the four base resistance values may be obtained from a least-squares fit of the values to the data. The four resistors need not be calibrated independently.

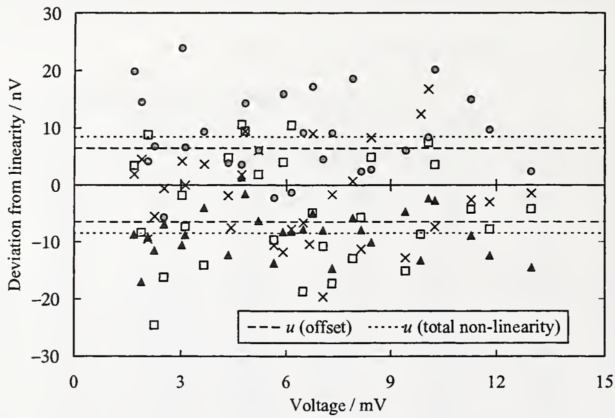

Figure 5. Tests of the non-linearity of a digital voltmeter at low voltage values. The four symbols represent four separate runs on different days.

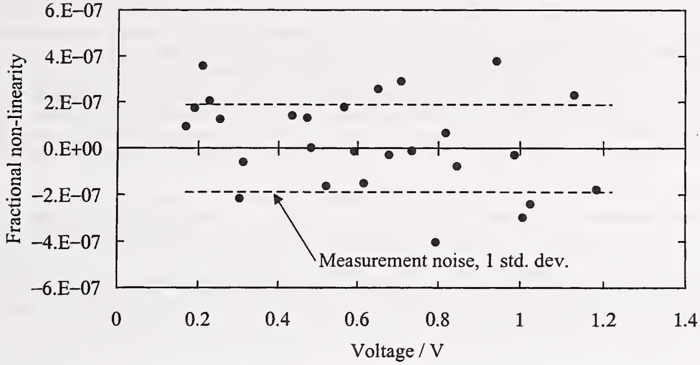

Figure 6. Test of the non-linearity of a digital voltmeter on the range with full scale voltage of 1.2 V.

Although the RBC is designed as an instrument for AC measurements or for DC measurements with frequent current reversals, it can be used with DC measurement systems provided that the thermal emfs in the RBC and its associated wiring are dramatically reduced. To reduce the thermal emfs of the wiring, it is necessary to use lead wires that are very similar in thermoelectric properties to the other lead wires and to the internal wiring of the digital voltmeter. This is accomplished by using twisted pairs of untinned copper wire instead of coaxial cable for the wiring. Thermal emfs inside the RBC are reduced dramatically by reducing the temperature gradients in the vicinity of the RBC. This is accomplished by thermally isolating the RBC inside concentric boxes of polystyrene foam, aluminum 0.8 cm thick, and polystyrene foam. The rms thermal emfs of the RBC connections when insulated in this fashion were less than 3.5 nV. In typical operation, the RBC is used to measure the non-linearity, offset, and gain error (these terms are defined in Section 10) of instruments that measure resistance ratios, the instrument reading being a measurement of the ratio of a test resistance to that of a reference resistance. For the purpose of measuring the non-linearity and offset of a voltmeter, a fixed current was passed through the RBC and an external, temperature-controlled reference resistor, both of which were connected in series. Resistance ratios were constructed by dividing the voltage measured across the RBC by the voltage measured across the reference resistor. The readings were corrected for the voltmeter zero readings either by averaging measurements with two directions of current, or by subtracting voltmeter readings made with zero current from the readings with current. As a consequence of using an external reference resistor that was not independently calibrated relative to the RBC base resistors, it was not possible to determine the voltmeter gain error with the RBC. The rms deviation of the readings from the simple model used to fit the RBC data is expected to equal the sum of the measurement noise and the voltmeter non-linearity, added in quadrature. Sources of measurement noise include both short-term fluctuations in the current source and the inherent noise of the digital voltmeter. At low voltages, the inherent voltmeter noise dominates the measurement noise, and each data point requires approximately seven minutes of signal averaging. By subtracting the independently-measured voltage noise from the rms deviation, we obtain an estimate of the non-linearity. The results of the RBC on the 100 mV voltmeter range indicate that the emf measuring system has a combined offset error (δ in Section 10) and non-linearity of only 0.0085 μV at the one standard deviation level, which we round to 0.01 μV. On the 1 V range, the offset is negligible, and the non-linearity is of the same magnitude as the measurement noise. Because we did not perform multiple runs on multiple higher voltage ranges, we chose to conservatively estimate the non-linearity on the higher ranges as the rms deviation from ideal behavior, without any subtraction of measurement noise. Previous measurements of the same model of voltmeter against a Josephson junction array gave similar results [16]. As shown in Fig. 6, the non-linearity is 2×10^{-7} of the reading, for readings of 16 % of nominal full scale and above.

We measured the gain error on the 10 V range directly by measuring a calibrated 10 V solid-state voltage reference. On the 100 mV range, which is the only range used for Au/Pt thermocouple measurements, it is possible to determine the gain error of the voltmeter by two simple methods. One straightforward method is to construct a

calculable 100 mV or 10 mV voltage source, based on a 1:1000 or 1:10,000 voltage divider constructed from two independently-calibrated four-terminal resistors that are connected in series. Passing a suitable DC current through the resistors produces voltages of nominally 10 V and 100 mV or 10 mV, whose exact ratio can be calculated from the resistor calibrations. A second method for determining the scale error relies on the high degree of linearity of the voltmeter, as shown in Fig. 5 and 6. If the voltmeter was perfectly linear, then the scale error of one range relative to another range can be determined by measuring the ratio of two determinations of the same voltage, on the two scales. For example, a 100 mV voltage can be read on both the 1 V scale and the 100 mV scale. In practice, an additional uncertainty must be added to account for the voltmeter non-linearity. We used this second method.

The voltmeter gain error (α in Section 10) is dominated by changes in the gain sensitivity of the voltmeter as the voltmeter temperature varies during the course of the day. Figure 7 shows variations in the ratio of the voltmeter readout to the true voltage, for a voltage of 100 mV, and as a function of the voltmeter internal temperature.

When these effects are combined, the Type B standard uncertainty for the combined scanner and digital multimeter system can be expressed as $u/\mu V = 2.5\times10^{-6}(E/\mu V) + 0.01$, where E is the thermocouple emf. A summary of the subcomponents of this expression is listed in Table 2.

Figure 7. Fractional deviation of measured voltage at 100 mV from value of voltage expected by scaling down from a 10 V reference, as a function of the temperature change from time of autocalibration.

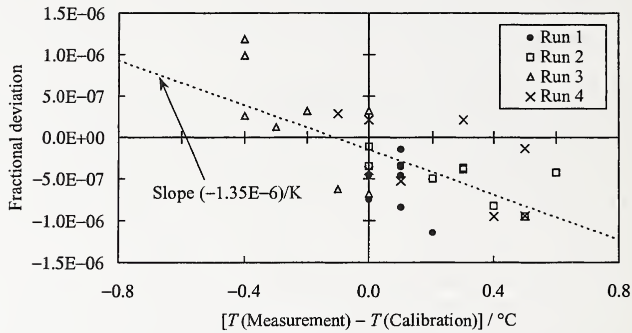

Table 2. Subcomponents of the gain uncertainty for the digital voltmeter, expressed as fractional standard uncertainty in E.

Subcomponent	Fractional standard uncertainty in E
Temperature change in voltmeter	1.2×10^{-6}
Voltmeter non-linearity	1.2×10^{-6}
Voltmeter calibration drift	1.0×10^{-6}
10 V dc voltage reference	1.5×10^{-6}
Combined gain uncertainty	2.5×10^{-6}

5.3 Realization of the ITS-90

For measurements at each fixed point, a Type B uncertainty was included to account for deviations of our cells from an ideal fixed point of a pure material. These deviations were determined by measurements of freezing plateaus with an SPRT or a high temperature SPRT, by comparison with the reference standard cells maintained by the Platinum Resistance Thermometry Laboratory at NIST, and by estimation of uncertainty from known impurities. In the data analysis, corrections were made to the fixed-point cell temperature for the hydrostatic head and for the temperature difference between the cells used for this study and the reference standard cells maintained in the Platinum Resistance Thermometry Laboratory.

5.4 Thermocouple inhomogeneity

As seen in the sample immersion profiles in Fig. 8 and 9, thermoelectric inhomogeneity will cause variations in the output emf of a thermocouple with changes in immersion depth even if the temperature of the measuring junction is fixed. Since it is unlikely that the SRM 1749 thermocouple will be used in a thermal environment identical to its calibration environment, an additional uncertainty u_i must be included to account for inhomogeneity. At the aluminum and silver fixed points, we have taken the standard uncertainty for this effect to be equal to the rms deviation of the measured emf values from the emf value at full immersion, for all immersions greater than 8 cm into the fixed-point cell:

$$u_i^2 = \frac{1}{n}\sum_{k=1}^{n}(E_k - E_0)^2 \quad , \qquad (1$$

where E_0 is the emf at full immersion, E_k is the emf at partial immersion, and n is the number of points taken at partial immersion. At other temperatures, we have estimated the inhomogeneity using linear interpolation and assuming that there is no inhomogeneity uncertainty at 0 °C.

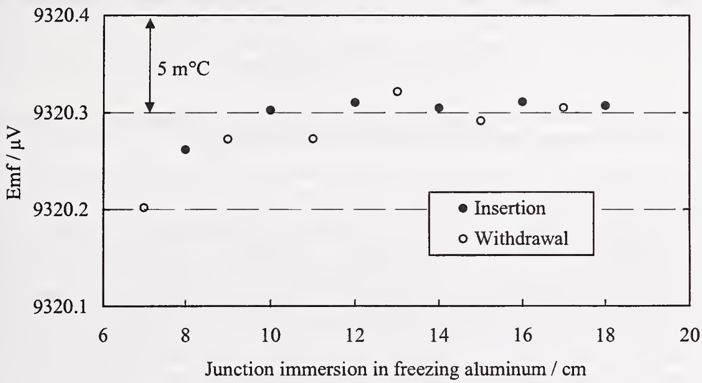

Figure 8. Values of emf measured for an SRM 1749 thermocouple on insertion into and withdrawal from the aluminum freezing-point cell during a freeze.

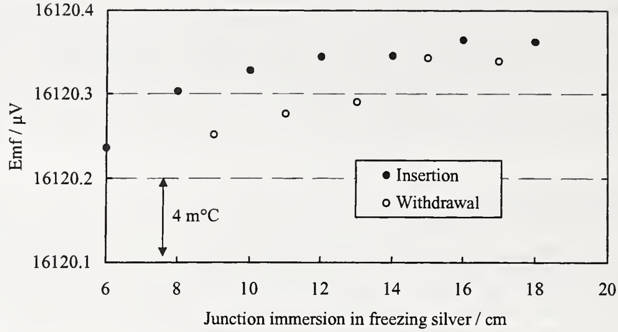

Figure 9. Values of emf measured for an SRM 1749 thermocouple on insertion into and withdrawal from the silver freezing-point cell during a freeze.

The stated inhomogeneity uncertainty, u_i, and combined expanded uncertainty, U, apply when the SRM 1749 thermocouple is used in apparatus where the immersion distance, L, from the coil of the measuring junction to the point at the throat of the furnace where there is a maximal thermal gradient is in the range 36 cm to 44 cm. For the two NIST furnaces used for all of the fixed-point measurements, this distance ranged from 38 cm to 44 cm. If the SRM 1749 thermocouple is used at an immersion significantly shorter than 36 cm from the point of maximum thermal gradient to the measuring junction, it is recommended that the uncertainty be recalculated according to the following formulas:

$$u_{i,L} = u_i + u_i (36\,\mathrm{cm} - L)/8\,\mathrm{cm} \quad , \tag{2}$$

$$U_L = \left(U^2 - u_i^2 + u_{i,L}^{\ 2}\right)^{1/2} \quad , \tag{3}$$

where $u_{i,L}$ is the standard uncertainty subcomponent for the thermocouple inhomogeneity at an immersion depth of L and U_L is the combined expanded uncertainty at the specified immersion depth.

5.5 Uncertainty of the reference function

In the calibration procedure, the true emf-temperature relation of the thermocouple is modeled as the reference function for Au/Pt thermocouples plus a quadratic function. This model is an approximation, with an associated uncertainty. Attempts to fit a cubic or higher order polynomial to the emf deviation data did not result in a statistically superior fit. If there are errors in the reference function that can be modeled with a quadratic function, such errors have no net effect on the uncertainty of the combined model of reference function plus quadratic function. This implies that the appropriate uncertainty is the uncertainty of the reference function over temperature ranges characteristic of the difference in temperature values of adjacent fixed points, which ranges from 75 °C to 301 °C. Because the number of degrees of freedom in the determination of the Au/Pt reference function was very high [7], the statistical uncertainty is generally small except at 1000 °C, and a good measure of the short-range uncertainty of the reference function is the systematic discrepancy between data obtained by fixed-point measurements and data obtained by comparison with an SPRT in the original determination of the reference function. This uncertainty for the Au/Pt thermocouple reference function is 0.04 µV at 1000 °C, 0.026 µV from the silver freezing point at 962 °C to the indium point at 157 °C, and decreases to approximately 0.014 µV for temperatures near 10 °C.

5.6 Uncertainty of the ice point

An ice point as prepared according to the methods described in Section 9 has a standard reproducibility of 1 m°C [17]. No uncertainty term has been added to account for the reproducibility of the ice point, since the Type A uncertainty for the reproducibility of the thermocouples emf at the various fixed points already incorporates any effects of variations in the ice points. However, we have added an uncertainty to account for any systematic errors in the NIST ice points caused by impurities in the distilled water used for the preparation of the ice and ice baths. Measurements of ice baths prepared from the same distilled water supply as used in the SRM 1749 calibration revealed a –0.6 m°C depression below the nominal value of 0 °C. The data has been corrected for this 0.6 m°C offset, and a square uncertainty distribution of ±1 m°C has been included for systematic variations in the ice point temperature.

Because of slight variations in the physical or chemical characteristics of the wires inside the reference-junction probe, the emf generated by the SRM 1749 thermocouples between room temperature and the reference junctions will vary slightly depending on the depth of immersion of the probe into the ice, even for immersion depths deep enough to ensure that the reference junctions are maintained at 0 °C. An uncertainty for this effect of reference probe inhomogeneity has been included to account for a variation of up to 1 cm in ice level or thickness of the reference bath cover in the user's laboratory compared to those at NIST. It is important to carefully adhere to the procedures of Section 9 to minimize these inhomogeneity effects. If different procedures are used, it is the user's responsibility to determine the effect on the thermocouple emf of using an ice point of different preparation or design. Preferably, this effect is determined by actual measurement of the emf differences obtained when the measuring junction is maintained at a fixed temperature and the bath used for the reference junction is alternated between the user's design and a bath prepared following Section 9.

5.7 Summary of uncertainties

As seen in Table 1 and Fig. 4, no single uncertainty component dominates the combined uncertainty over the full temperature range. At low temperatures, the uncertainty of the reference function is dominant. Above 962 °C, the reference function is based on an extrapolation of the ITS-90, which is not well known, and this becomes the dominant uncertainty. For intermediate temperatures, the thermocouple reproducibility, the uncertainty of the emf measurements, and the thermocouple inhomogeneity are all significant.

6. Properties of thermocouples prepared for SRM 1749

The emf deviation, defined as the difference between the measured emf of a particular thermocouple and the reference thermocouple, is a convenient means of comparing the emf-temperature relation of a thermocouple to the reference function. Figure 10 shows the average, maximum, and minimum emf deviation for the lot of SRM 1749 thermocouples. The maximum deviation from the reference function for all 18 thermocouples in the lot is 0.22 μV at the aluminum freezing point, equivalent to 11 m°C. The maximum spread in the emf values at any of the fixed points is 0.21 μV at the silver freezing point, equivalent to 8.5 m°C, which is approximately a factor of 10 smaller than we observe with platinum-rhodium alloy thermocouples fabricated from the same lots of wire. The small spread in the emf values is an indication that the gold and platinum wires used to fabricate the thermocouples were chemically homogeneous, and that the annealing procedures used in the fabrication process were highly reproducible and uniform.

The scatter at each fixed-point temperature shown in Fig. 10 is not random. Correlation plots of the emf deviation at one fixed-point temperature versus the emf deviation at another fixed-point temperature show a high degree of correlation for the emf measurements at the lower-temperature fixed points. This can be seen in the representative plots in Fig. 11 and 12. At the higher temperatures, there are correlations in the emf readings, but the correlation is relatively weak compared to the thermocouple reproducibility, as seen in Fig. 13. This result confirms that there are statistically significant differences in the emf-temperature relationships of the 18 thermocouples in the lot at lower temperatures and that individual calibration gives a smaller combined uncertainty than using an average calibration for the lot.

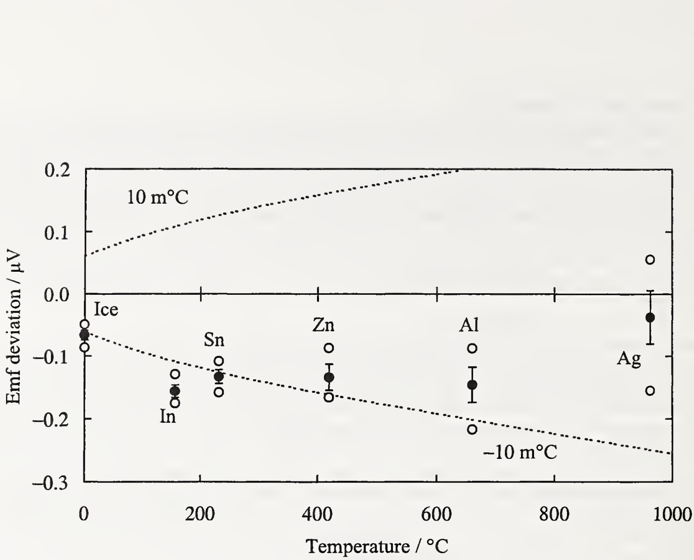

Figure 10. Differences between the measured emf values for the SRM 1749 thermocouples and the reference function for Au/Pt thermocouples. Solid circles indicate the average values for all thermocouples, the open circles indicate maximum and minimum values, and the error bars indicate one standard deviation variations in the emf values.

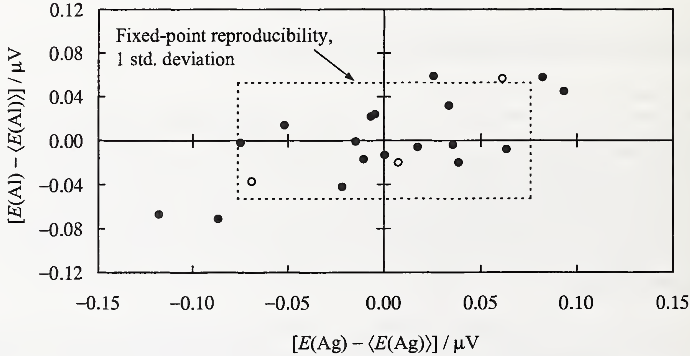

Figure 11. Correlation of emf values at the aluminum point, *E*(Al), with emf values at the silver point, *E*(Ag). Solid circles are for the SRM 1749 thermocouples. Open circles are from additional thermocouples fabricated from the same lots of wire as the SRM 1749 thermocouples.

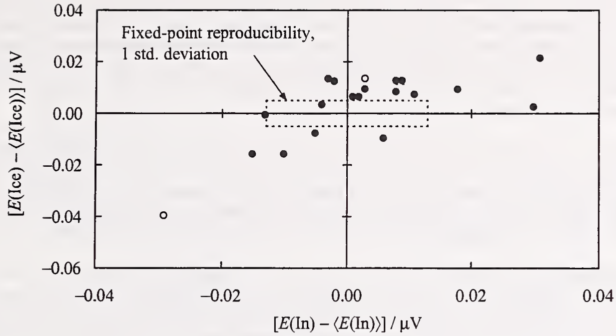

Figure 12. Correlation of emf values at the ice point, *E*(Ice), with emf values at the indium point, *E*(In). Solid circles are for the SRM 1749 thermocouples. Open circles are from additional thermocouples fabricated from the same lots of wire as the SRM 1749 thermocouples.

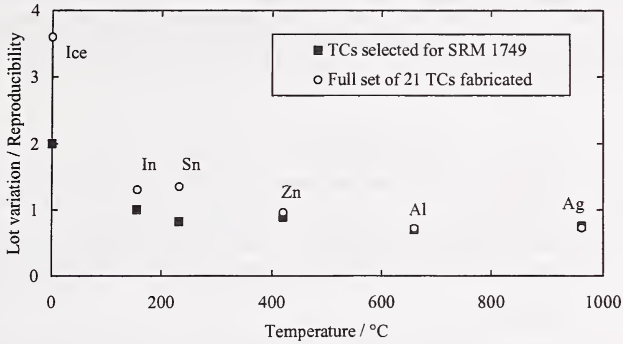

Figure 13. Ratio of the standard deviation of emf values in the lot of SRM 1749 thermocouples at each fixed point to the standard deviation for the reproducibility of Au/Pt thermocouples with extended thermal cycling.

Because the Seebeck coefficient is quite small near 0 °C, over a factor of four smaller than at 1000 °C, the emf deviations of the SRM 1749 thermocouples at the ice point are equivalent to relatively large temperature deviations. The average deviation in units of temperature is −11 m°C at 0 °C. This result confirms the necessity of including the ice point as a calibration point.

Figures 8 and 9 show representative immersion profiles at the aluminum and silver fixed-point cells. These results are very typical of high-quality Au/Pt thermocouples, showing little variation in emf values beyond an immersion of 8 cm of the measuring junction below the surface of the freezing metal. At the silver freezing point, there is noticeable hysteresis between measurements taken on insertion of the thermocouple into the cell, compared to measurements taken on withdrawal. This is most probably a result of reversible metallurgical changes, such as a

change in the number of lattice vacancies in the thermoelements, or possibly changes in the oxidation state of the platinum. When annealed at 450 °C for 10 h, the metallurgical state of the thermoelements attains thermal equilibrium. On subsequent use at higher temperatures, the sections of the thermoelements exposed to significantly higher temperature will have an increased number of lattice vacancies. As the thermocouple is first inserted into and then withdrawn from the furnace, the thermoelements will acquire a nonuniform distribution of lattice vacancies along the thermoelement length.

Measurements were also made of the sensitivity of the thermocouple emf to immersion of the reference-junction probe. These measurements showed that immersion of at least 20 cm into the ice/water mixture was sufficient to assure thermal equilibrium of the reference junctions with the ice/water mixture. On insertion of a reference-junction probe from room temperature into an ice bath, it is necessary to wait ten minutes for the reference junctions inside the probe to reach a steady state temperature, within the uncertainty of the calibration.

Over 1000 h of use at temperatures up to 963 °C, a Au/Pt thermocouple will maintain this stated accuracy. Figure 14 shows the emf values at the silver freezing point of one Au/Pt thermocouple that has been used as a check standard at NIST. The data shown were obtained from measurements of 105 freezes of the silver freezing-point cell from February of 1995 to April of 1997. No long term drift has been observed with several Au/Pt thermocouples that have been studied for periods of time exceeding 1000 h and that have been maintained in accordance with the procedures of Section 5.

Measurements on one of the SRM thermocouples at the silver freezing point confirmed that the special construction of the SRM 1749 thermocouples did not impair this long term stability. The thermocouple, with its silica glass sheath attached, was aged at a temperature of 980 °C for a cumulative period of 800 h. Periodically during this process, the thermocouple was measured at the silver freezing point. The results of this test are shown in Fig. 15. The rms deviation of all measured points from the initial measurement at the silver point is only 0.055 μV, equivalent to 2.2 m°C, which is less than the standard reproducibility of 2.8 m°C and significantly less than the combined standard uncertainty of 4.2 m°C at this temperature

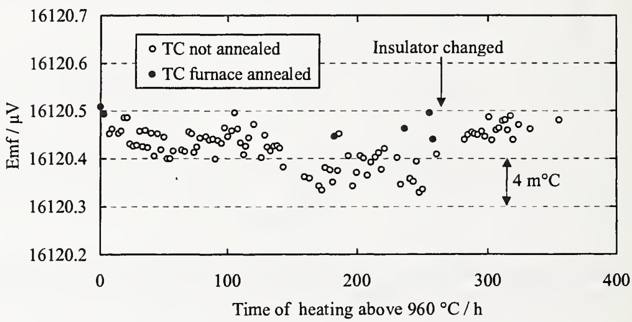

Figure 14. Emf value at the silver freezing point of a thermocouple fabricated at the same time and from the same wire lots as the SRM 1749 thermocouples, and used as a check standard.

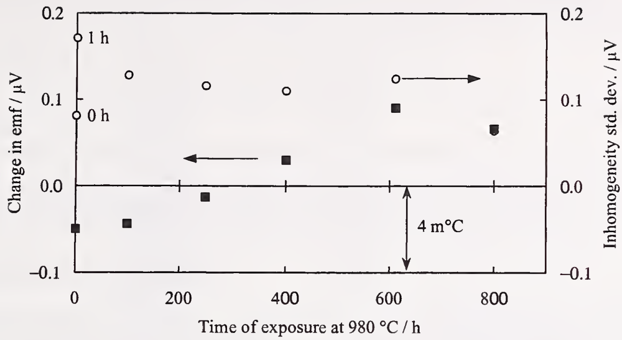

Figure 15. Inhomogeneity and change in the emf measured at the silver freezing point for an SRM 1749 thermocouple that was heated at 980 °C.

7. Mathematical methods for use with SRM 1749

The standard emf tables supplied with the SRM 1749 thermocouples have a resolution of 0.1 μV. This resolution is equivalent to a temperature resolution as poor as 17 m°C at 0 °C, which is considerably worse than the calibration uncertainty of the thermocouples. For the highest accuracy, it is necessary to use the polynomial calibration function

$$E = a_0 + \sum_{i=1}^{N} a_i t_{90}{}^{i} \quad , \qquad (4$$

with the coefficients that are supplied in Table 3 in the SRM 1749 Certificate. An example of these coefficients is given in Table 3. To determine the emf value of the SRM 1749 thermocouple at a particular temperature, straightforward evaluation of the calibration function suffices. The software used must perform the calculations with an accuracy of at least eight digits. Table 4, which includes a set of temperature and emf data pairs out to a resolution of the equivalent of 0.1 m°C evaluated from the coefficients in Table 3, may be used to check the validity of user software.

Table 3. Sample coefficients, a_i, of a calibration function for SRM 1749 thermocouples. (each SRM 1749 thermometer is supplied with unique values of a_i.) The function gives the thermoelectric voltage, E, in millivolts, as a function of temperature, t_{90}, on the ITS-90 for the temperature range 0 °C to 1000 °C.

Coefficient	
a_0	$-1.050\,000\,00 \times 10^{-4}$
a_1	$6.035\,698\,61 \times 10^{-3}$
a_2	$1.936\,759\,74 \times 10^{-5}$
a_3	$-2.229\,986\,14 \times 10^{-8}$
a_4	$3.287\,118\,59 \times 10^{-11}$
a_5	$-4.242\,061\,93 \times 10^{-14}$
a_6	$4.569\,270\,38 \times 10^{-17}$
a_7	$-3.394\,302\,59 \times 10^{-20}$
a_8	$1.429\,815\,90 \times 10^{-23}$
a_9	$-2.516\,727\,87 \times 10^{-27}$

Table 4. Temperature-emf data pairs evaluated from the coefficients in Table 3, to a resolution of the equivalent of 0.1 m°C. These data pairs may be used to check the validity of user software.

t_{90} / °C	E/mV
0	−0.000 105 0
100	0.777 746 3
200	1.844 884
300	3.141 542
400	4.633 170
500	6.300 671
600	8.134 800
700	10.131 941
800	12.290 580
900	14.609 001
1000	17.085 005

Table 5. Coefficients c_i of an approximate inverse function for Au/Pt thermocouples. This function gives temperature, t_{90}, in degrees Celsius as a function of the thermoelectric voltage, E, in millivolts in the specified temperature and voltage range. The error of this inverse function is ±1 °C. See Eq. 5. Note that the error of this inverse function is much larger than the error of the inverse function in Ref. [18], which is somewhat more mathematically complex. The function below should be used only in conjunction with the method of section 7.

Coefficient	
c_0	0.000 000
c_1	$1.545\ 738\times10^{2}$
c_2	$-4.219\ 267\times10^{1}$
c_3	$1.351\ 401\times10^{1}$
c_4	−2.888 146
c_5	$3.931\ 653\times10^{-1}$
c_6	$-3.366\ 290\times10^{-2}$
c_7	$1.751\ 092\times10^{-3}$
c_8	$-5.047\ 341\times10^{-5}$
c_9	$6.176\ 037\times10^{-7}$

To determine the temperature value of the SRM 1749 thermocouple at a measured emf value requires a more complex method. Because there is no exact inversion of the polynomial calibration function, a numerical inversion technique must be used. The steps of this procedure are as follows:

1. Evaluate the approximate inverse temperature t_{app}, using the inverse function

$$t_{\mathrm{app}} = c_0 + \sum_{i=1}^{N} c_i E_{\mathrm{m}}{}^{i} \quad , \qquad (5$$

where the coefficients c_i are listed in Table 5 and E_{m} is the measured value of emf in units of millivolts. (The approximate inverse equation given in Ref. [18] may also be used. This alternate function gives a somewhat more accurate initial value for t_{app}, but it is only the tolerance specified in step 5, and not the choice of approximate inverse function, that determines the accuracy of the final value of t.)

2. Define the function $G(t) = E_{\mathrm{m}} - E_{\mathrm{cal}}(t)$, where $E_{\mathrm{cal}}(t)$ is the SRM 1749 calibration function given in Table 3 of the Certificate.
3. The derivative of $G(t)$ is given as

$$G'(t) = -\sum_{i=1}^{N} i a_i t^{i-1} \quad . \qquad (6$$

4. Use the iterative technique termed Newton's method to find the value of t that will give $G(t) = 0$. This value of t is the desired temperature value corresponding to the measured emf value. For each iteration of Newton's method, an improved temperature value, t_1, is determined from a starting value of temperature, t_0, using the equation:

$$t_1 = t_0 - G(t_0)/G'(t_0) \quad . \tag{7}$$

For the first iteration, $t_0 = t_{app}$. For later iterations, the value of t_0 is taken as the value of t_1 determined in the previous step.
5. Repeat iterations of Newton's method until the change $|t_0 - t_1|$ is less than a tolerance set by the user.

In practice, only one or two iterations will be necessary, since all of the SRM 1749 thermocouples are a very close match to the reference function. Again, for checking user software, Tables 3 and 4 are useful.

8. Maintenance of the thermocouple

Pure element thermocouples are fairly rugged devices, capable of withstanding repeated thermal cycling and small mechanical shocks. However, three maintenance procedures are necessary to ensure that there is no degradation in accuracy.

First, the platinum expansion coil at the measuring junction should be inspected periodically. At room temperature, both thermoelements should extend from the alumina tube by the same amount as when the thermocouple was first manufactured. If the protrusion of one of the thermoelements increases by more than about 1 mm after a period of heating to elevated temperatures and then cooling to room temperature, the thermoelement may have seized slightly in the bore of the alumina tube and may have been mechanically strained.

Second, pure element thermocouples should be given a periodic maintenance anneal especially if they are removed quickly from high temperature environments or if they are used in different thermal environments. The procedure for Au/Pt thermocouples is to furnace anneal the thermocouple at 450 °C overnight at the fullest possible immersion of the thermocouple into the furnace. This procedure reduces the number of lattice vacancies quenched into the thermoelements when the thermocouple is removed rapidly from a high-temperature environment. At NIST, the furnace used for furnace annealing is a three zone, horizontal tube furnace. During a heat treatment, the temperature along the portion of the thermocouple extending from its measuring junction to approximately 62 cm from the junction was uniform within 2 °C.

Third, the copper leads of the SRM 1749 thermocouple are not plated, and will become oxidized with time. Although the use of a junction box similar to the one described in Section 10 will minimize the thermal gradients near the terminals, it is still recommended that the copper oxide be removed occasionally by pulling the short, exposed sections of bare copper lead wire gently through a folded abrasive pad. With extensive use, these copper wires may become work-hardened and fragile. In this event, the user may cut off the short affected length of copper wire and strip off a short section of wire insulation without affecting the calibration of the thermocouple.

9. Preparation of an ice bath for the reference junctions

The low uncertainty of the SRM 1749 thermocouples requires a carefully prepared ice bath to maintain the temperature of the reference-junction probe at 0 °C. A properly prepared ice bath will have an expanded uncertainty of 2 m°C.

The ice for the ice bath should be finely-crushed or shaved ice that has been prepared from distilled water. The ice should be saturated with distilled water, and then packed gently into an insulating Dewar flask, such that ice fully fills the volume of the flask with no large voids. At NIST, a cylindrical flask (7 cm inner diameter and 30 cm deep) having a polyethylene-foam cover, 2.5 cm thick, is used. Other flask geometries are allowable, provided the flask is at least 6 cm inner diameter and 30 cm deep, and the thickness of the cover is in the range 1.5 cm to 3 cm. The level of the ice-water mixture should be within 5 mm of the bottom surface of the cover. The cover should have a hole of approximate diameter 6.3 mm in the center, to allow insertion of the reference-junction probe of the SRM 1749 into

the ice point. The reference-junction probe should be inserted until the stop on the probe is butted against the flask cover. Since the ice in the Dewar flask will tend to float as the ice melts, a rubber band should be used to secure the cover onto the flask.

The use of electronic ice-point compensators, extension wires, and automated ice points cooled with thermoelectric modules is not recommended unless a careful analysis of the additional uncertainties is performed. These devices, in general, contribute additional errors to the measurement that are large relative to the calibration uncertainty of the SRM 1749.

It is possible to use a triple point of water (TPW) cell to maintain the reference junctions at a temperature of 0.01 °C. If this is done, a quantity of 0.056 μV should be added to the measured emf to compensate for the reference temperature differing from the nominal 0 °C. The enclosure used to maintain the TPW cell should be configured so that the thermal profile along the reference-junction probe of the SRM 1749 is similar to the profile that the probe would be exposed to in a ice bath as described above.

10. Emf measurements

The calibration uncertainty of the SRM 1749 thermocouples, when expressed in units of voltage, ranges from an expanded uncertainty of 0.04 μV at a thermocouple signal of 0 μV (0 °C) to 0.37 μV at a thermocouple signal of 17,085 μV (1000 °C). In comparison, many voltmeters have expanded uncertainties significantly greater than this. In order to achieve the inherent accuracies possible with the SRM 1749 thermocouple, it is necessary to select the equipment for the voltage measurements carefully and to take the measurements using proper procedures. This section describes considerations for equipment selection and describes the methods used at NIST to measure voltage.

Although it is possible to connect the SRM 1749 thermocouple directly to a digital voltmeter, lower measurement uncertainties and greater convenience are possible by connecting the digital voltmeter to a scanner, which is an instrument that allows sequential connection of a voltmeter (or other instrument) to any one of a number of voltage sources. A single set of terminals to be used for reading one voltage source consists of a positive-polarity terminal, a negative-polarity terminal, and possibly a guard terminal, and is commonly termed a "channel" on the scanner. Because the terminals for each channel are often not conveniently placed for attaching leads of the thermocouple, it is desirable to extend the terminal connections of the scanner to a specially-constructed junction box. Consequently there are three pieces of equipment needed for high accuracy voltage measurements: a digital voltmeter, a scanner, and a junction box. Each of these items will be considered below, and then the data acquisition procedures to be used for the combined system will be described.

10.1 Digital voltmeters

Manufacturers typically state the specifications for DC voltage measurements with a digital voltmeter as a fraction of the voltage E that is measured, plus a fraction of the range. For example, on a 100 mV range, the specifications may be quoted as 4H10^{-6} of reading plus 3H10^{-6} of range, which is mathematically expressed as a tolerance a =(4H$10^{-6}E$ + 0.3 μV). If the user does no further characterization of the voltmeter, then the standard uncertainty contributed by the voltmeter may be estimated by assuming that the manufacturer's tolerance sets bounds $\forall a$ for a rectangular distribution of measurement errors. The standard uncertainty of such a distribution is $a/\sqrt{3}$, or for the example above, $u(E)$ = (4H$10^{-6}E$ + 0.3 μV)/$\sqrt{3}$.

In practice, we have found that it is possible with proper measurement techniques to greatly reduce the component of $u(E)$ that is independent of E. To achieve a smaller value of $u(E)$ it is necessary to understand the various effects that can cause voltmeter measurement uncertainties. The sources of voltage measurement errors include the following effects:

1. Gain error of the voltmeter, resulting in $R = (1 + \alpha)E$, where R is the reading of the voltmeter and α = constant. Although α is independent of E, it will in general vary with the temperature of the voltmeter and may change over periods of time from several days to several months.

2. Offset error of the voltmeter, resulting in $R = E + \delta$. Typical sources of δ are differences in the thermal emfs inside the voltmeter relative to the thermal emfs that existed when the voltmeter was calibrated, and possibly other noise contributions that vary at such a low frequency that signal averaging is ineffective. The value of δ varies over time scales ranging from several minutes to several months.
3. Nonlinearity of the voltmeter, resulting in higher order terms in the $R(E)$ relation than can be modeled with a simple linear relation. In most modern voltmeters, the non-linearity is of the same order as the magnitude of the least significant digit.

Of these three components, only the offset error is amenable to reduction by the user. Non-linearity is difficult to measure and tends to be a small effect in any case. The gain error in general is different for each measurement range and may vary substantially as the internal temperature of the voltmeter varies. One could correct for gain error by measurements of a reference voltage every hour or so on the same range as the thermocouple measurements, but it is difficult to build or obtain a reference voltage of sufficient accuracy at the low voltages generated by thermocouples.

Fortunately, the offset error is often a large contribution to the overall uncertainty, and demonstrated reduction in the offset error can justify a reduction in $u(E)$. A procedure that is effective in both reducing the offset error of the voltmeter and in correcting for thermal emfs contributed by the scanner is described in the Sections 10.2 and 10.4. In selecting a voltmeter for use with the SRM 1749, there is no perfect choice. So called "nanovoltmeters" have low offset errors but relatively large gain errors. These units are ideal when the manufacturer's specifications for the term in the DC voltage accuracy proportional to E is acceptably low. Other voltmeters without input stages optimized for low voltages, but with a high number of significant digits in the readout, typically have larger offset errors and greater noise, but substantially smaller gain errors than nanovoltmeters. With careful measurement techniques and with sufficient signal averaging to reduce the measurement noise, this type of meter has given the most accurate results at NIST.

10.2 Scanners

At NIST, we have had success with a variety of scanners that have been optimized for low-level DC voltage measurements. Some scanners have been designed with extremely low thermal emfs (less than 50 nV), and these are highly appropriate for use with the SRM 1749 thermocouple. Surprisingly, scanners with specifications that are significantly less stringent will serve well provided that the scanner is appropriately selected and that the data acquisition procedure described below is followed. As a general rule, avoid scanners that route analog signals via multipin connectors, which can be a significant source of extraneous thermal emf. As an example of a connector type that should be avoided for measurements of the highest accuracy, some scanners require that the signal leads be terminated at a multipin connector, which then is plugged into a mating connector on a circuit board of the scanner. The signal path from the thermocouple should pass through only screw connectors, copper wires or circuit board traces, and the relays. Multipin connectors that provide power or that route digital control signals are acceptable. If the scanner uses electromechanical relays that are continuously energized while a particular channel is being read, the heat from the relay coil may cause significant changes in the thermal emfs. This effect can be minimized by a) energizing each relay for no more than 1 minute at a time, b) using each relay at a duty cycle of no more than 20%, and c) setting the scanner to a specially-designated "hold" channel when data is not being acquired. To achieve the low duty cycle while still utilizing all available measurement time, it is convenient to route the thermocouple signal to several pairs of terminals by using bare copper wires between terminals on the junction box.

Even with the above precautions, we have found that most commercial scanners still have extraneous thermal emfs that are unacceptably large for use with the SRM 1749 thermocouple. These emfs are relatively reproducible, though, and it is straightforward to correct for them. The procedure is as follows:

1. Each channel of the junction box is shorted with bare copper wire, the top of the box is covered with a layer or box of insulating foam to reduce temperature gradients, and the wiring is allowed to come to thermal equilibrium for 15 minutes.
2. One channel, or a set of channels, is designated as the "short" channel. The copper shorting wires will remain on these channels for all measurements, including measurements of the thermocouple emf. The "short" channel or channels is the reference against which other channels are compared.
3. Using the identical software used for acquisition of thermocouple emf data, each of the junction box channels is measured several times.

4. The readings for each channel are averaged. The difference between the measured emf of each channel and the average of the short channel(s) is computed. This difference is then used to correct all later readings on that channel. For example, assume that channel 1 will be the short channel and channel 2 will be used for the thermocouple readings. If channel 2 reads an average of 0.1 μV higher than channel 1, when both are shorted, then subsequent readings of the thermocouple on channel 2 should be reduced by 0.1 μV to compensate for this difference.
5. Note that the absolute value of voltage read on each channel is not used in the computation of this channel correction. Adjustment of the data for a voltage reading on the short channels not equaling zero will be discussed below.

It is recommended that this procedure be done several times, at different times during the day, to evaluate the fluctuations in the channel corrections. A plot of the channel corrections versus time will show random fluctuations due to measurement noise and possibly drift over periods of several hours or days due to changes in the extraneous thermal emfs of the wiring and scanner card. The frequency of the measurement of the channel corrections can be determined from the observed drift rate of the corrections. At NIST, to achieve the highest possible accuracy, we determine the corrections as often as twice a day.

Connection of the scanner to a junction box and the construction of junction boxes are discussed in the next section.

If the scanner has mechanical relays, circuit heating by the relay coil may be minimized by closing the relay on each channel only long enough to make the measurement, then proceeding to a different channel. If there is an extended period between measurements, the scanner may be set to a channel that is dedicated to monitoring purposes only, and that is never used in the acquisition of calibration data. When evaluating a new scanner, it is also desirable to test all scanner channels by placing copper wire shorts across each set of terminals on the isothermal block, and then measuring the emf for each channel. Channels that give outlying values of emf should not be used, or used for non-critical purposes.

In regular use, one of the scanner channels should remain shorted at the isothermal box. Periodic readings of the voltmeter on this channel will allow correction for the voltmeter "zero". This procedure is superior to use of any "null" button on the voltmeter because the readings of the voltmeter on the shorted channel can be monitored.

10.3 Junction boxes and wiring

The isothermal junction boxes used at NIST consist of an aluminum box with a set of labeled terminals on top of the box. The terminals are assemblies of gold-plated copper washers and nylon screws that fasten into a 6.4 mm aluminum plate. One of the washers for each screw is a specially made washer with a "pigtail" that passes through an insulated hole in the aluminum plate. Each of the labeled terminals is connected to the corresponding screw terminal inside the scanner, using bare copper wire insulated with polyvinylchloride polymer. For the NIST boxes, the copper lead wires are fastened to the pigtails with low thermal emf solder (Cd-Sn eutectic). However, cadmium vapors generated during soldering are quite toxic and Cd-Sn is hard to obtain. Alternatively, the connection between the terminal and the wire can be made by one of three techniques: a) solder with pure indium, b) establish a tight mechanical connection by clamping between two nuts, or c) establish a tight mechanical connection by crimping a soft pure copper tube over the terminal and wire. The thermal emf of indium is quite low relative to copper, and so the soldered joints will not generate significant thermal emf. (However, copper-indium joints may become weak over many years due to atomic diffusion at the interface.)

Wiring from the scanner to the junction box is fabricated from bare copper wire insulated with polyvinyl chloride polymer. Such wire is sold as telephone wire or as specialized wiring for computer network applications. For immunity from electromagnetic pickup, the wires for the positive and negative lead of each channel should be a twisted pair. It is possible to wire the junction box using the multicolored twisted pairs of the as-supplied wire, but if the scanner terminals in use are at a hotter temperature than ambient, small differences in the copper compositions of the different-colored wires will result in small extraneous thermal emfs. At NIST, all scanners with terminal temperatures more than several degrees hotter than ambient are wired with twisted pairs fabricated from a single length of wire folded back on itself and then twisted. Fabrication of this twisted pair and the work of identifying the positive and negative leads of each pair are time consuming, but the reduction in stray thermal emf is significant.

The terminals on the top of the box are designed to generate minimal thermal emfs. At NIST, we use home-made terminals made of gold-plated copper, secured to the aluminum top with nylon screws and insulated from the top with nylon washers. Commercial binding posts made of tellurium copper will also suffice. The aluminum top itself is either painted or anodized to prevent electrical shorts by accidental contact of thermocouple wires with the top of the box. The aluminum top is approximately 6 mm thick, to provide effective shunting of any thermal gradients in the vicinity of the box. A schematic diagram of a junction box is shown in Fig. 16.

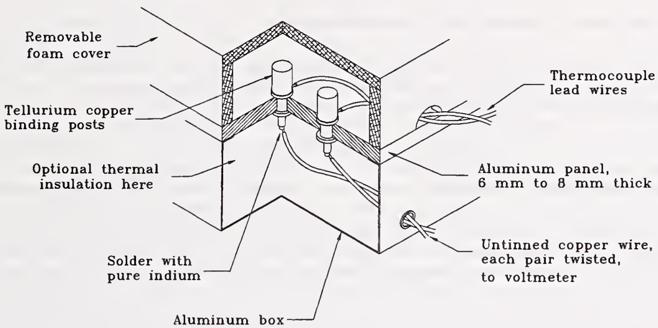

Figure 16. Schematic cross section of a junction box with low thermal emfs, constructed with commercial binding posts.

10.4 Methods for data acquisition

For the highest accuracy results, emf measurements should be taken using an automated system. This is especially important in two cases: 1) when using voltmeters that have not been optimized for very low voltage measurements, automated signal averaging of multiple measurements is necessary to reduce the measurement uncertainty, and 2) when a scanner with electromagnetic relays is used, any relay heating is predictable and can be corrected for, provided that the data acquisition is automated to give highly repeatable timing of the data acquisition. At NIST, we have achieved excellent results by setting the internal filters or time constants of the voltmeter at an equivalent time constant of approximately 0.5 s, and then performing additional averaging of the data after computer acquisition.

An example of a typical data acquisition cycle follows:

Channel 1	Short, 10 readings spaced at 0.5 s intervals
Channel 3	Thermocouple, 10 readings spaced at 0.5 s intervals
Channel 4	Thermocouple, 10 readings spaced at 0.5 s intervals
Channel 5	Thermocouple, 10 readings spaced at 0.5 s intervals
Channel 8	Short, 10 readings spaced at 0.5 s intervals

Repeat above cycle five times

Channel 12	Test thermocouple ... no calibration data acquired on this “hold” channel

After each channel is selected, there is a short wait (less than one second) for the voltages to settle, and then 10 readings are acquired at 0.5 s intervals. Note that some channels may not be used (channels 2, 6, and 7 above), after determination by the procedure in Section 10.2 that some channels have larger than desired emf corrections or poor repeatability of the emf corrections. The parallel connection of one test thermocouple to multiple channels enables almost continuous data acquisition while minimizing the self-heating of any one channel. Additionally, any errors

in the determination of the scanner emf corrections is averaged out somewhat. Once a data acquisition procedure has been chosen, the same procedure should be used to measure the scanner emf corrections as is used to acquire the thermocouple emf data.

Following acquisition of the data from the cycle above, the scanner emf corrections are applied and the average of the "short" channels is subtracted from the readings on the "thermocouple" channels. The resulting values are corrected both for offset errors of the voltmeter and for emf offsets of the scanner channels.

11. Preventing thermocouple contamination

At high temperatures, metal ions can readily diffuse through silica glass and metals or metal oxides can become volatile. If the gold or platinum thermoelements of the thermocouple are contaminated by other metals, significant degradation of the thermocouple properties will occur.

Unlike thermometers such as platinum resistance thermometers, a thermocouple generates emf only in regions where the thermoelements pass through a thermal gradient. In proper usage, the measuring junction of a thermocouple is placed in a relatively isothermal environment, deep inside a uniform zone of a furnace or isothermal block, for example. As a consequence of the small thermal gradients, the portion of the thermocouple near the measuring junction contributes only a small portion of the total emf generated by the thermocouple, and contamination of the measuring junction region of the thermocouple will have negligible effect. In contrast, it is very important to protect from chemical contamination the portion of the thermocouple that passes from room temperature to a temperature close to that of the measuring junction. Experience with type S thermocouples has shown that sintered alumina provides an effective barrier to thermocouple contamination, provided that the alumina is not cracked. We do not recommend that the inner alumina insulator of the SRM 1749 thermocouple be directly inserted into environments that could potentially contaminate the alumina. Instead, the supplied glass sheath or a customer-supplied alumina or glass sheath should be used when inserting the thermocouple into high-temperature metal blocks or similar environments.

For Au/Pt thermocouples mounted in two-bore alumina insulators and then placed in a large diameter, high-purity alumina tube, we have seen no evidence of drift after 1000 h of heating at 962 °C. For Au/Pt thermocouples mounted in two-bore alumina insulators and then placed in a silica glass tube that is surrounded by a copper isothermal block, we have seen no evidence of drift after approximately 100 h of heating at 1000 °C.

If the Au/Pt thermocouple is used in an Inconel equilibration block with a silica glass sheath around the thermocouple, the drift in the thermocouple should be no worse than the case above with the copper block, since copper is both more volatile and more mobile than the constituents of Inconel.

12. Comparison measurements with the Au/Pt thermocouple

A common application of a Au/Pt thermocouple is to calibrate a second thermometer by comparison against the Au/Pt thermocouple. The success of this measurement requires careful attention to 1) the rate of change of temperature with time and 2) the effects of temperature non-uniformity.

If the temperature of the furnace or bath used for the calibration varies with time, there are two effects:

First, the diffusion of heat into (or out of) the measuring junction of the Au/Pt thermocouple and into the test thermometer is not instantaneous, and there will be dynamic temperature gradients in the vicinity of both thermometers, in addition to the static gradients that exist when the furnace is at a constant temperature. This effect can be measured by performing calibrations at different temperature drift rates of the furnace or bath. Data should be obtained for both rising and falling temperatures. A plot of the temperature reading of the Au/Pt thermocouple minus the reading of the test thermometer versus temperature drift rate will indicate the sensitivity of the calibration to the drift rate. The sensitivity to temperature drift will depend not only on details of the furnace or bath environment, but also on the thermal characteristics of the test thermometer.

Second, if the test thermometer and the Au/Pt thermocouple are read sequentially instead of simultaneously, there will be an error equal to the furnace drift rate multiplied by the time difference of the two readings. When the temperature is changing approximately linearly with time, this second effect can be minimized by taking readings in the order: Au/Pt thermocouple, test thermometer, Au/Pt thermocouple at equal time intervals. Averaging the two readings of the Au/Pt thermocouple gives the effective thermocouple reading at the time of the reading of the test thermocouple.

If the temperature drift of the furnace or bath is zero, the temperatures of the Au/Pt thermocouple and the test thermometer will still differ, in general, because of static temperature gradients in the furnace. We have had good results using Au/Pt thermocouples as reference thermometers in stirred-liquid baths of a variety of types. Use of Au/Pt thermocouples as a reference thermometer in a furnace is much more problematic, since typical spatial temperature variations in a furnace can be much higher than the uncertainty of Au/Pt thermocouples.

To attain the highest accuracy possible in calibrating a test thermocouples against a Au/Pt thermocouple in a furnace, a measuring junction common to all of the thermocouples is formed. Unfortunately, this method is labor intensive, requires skillful manipulation of the thermocouple wires, and can only be used in the calibration of other noble metal thermocouples. The method should only be attempted by those highly skilled in welding noble metal wires. First, any expansion coils or measuring junctions are removed. Second, each thermoelement is extended by welding to it a 1.5 cm length of high purity, 0.12 mm diameter wire of the same nominal composition as the thermoelement. Finally, the ends of these fine wires are welded together to form a common measuring junction, leaving sufficient slack in the wires to minimize the effects of the different thermal expansions of the three metals. The common measuring junction ensures that the measuring junctions of all thermocouples are at virtually the same temperature. This method has been used in the determination of a highly accurate reference function for Pt/Pd thermocouples, for example [8].

An alternate method of reducing temperature gradients is to place an isothermal block inside the furnace. Such blocks are commercially available, fabricated out of Inconel, graphite, or nickel. Prior to use in regular calibrations, the performance of an isothermal block should be tested by performing calibrations at varying rates of heating and cooling, and by interchanging positions of test and reference thermocouples, and by varying the immersion depths of the test and reference thermocouples.

At the time of preparation of this document, there is little guidance available on the performance of various isothermal block designs with Au/Pt thermocouples. This is an active area of research.

13. References

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National Institute of Standards & Technology

Certificate

Standard Reference Material® 1749

Gold versus Platinum Thermocouple
Certified Thermometer for the Range 0 °C to 1000 °C
on the International Temperature Scale of 1990

Serial No.

This Standard Reference Material (SRM) is intended for use as a highly accurate secondary reference thermometer in the range from 0 °C to 1000 °C [1]. SRM 1749 consists of a specially constructed gold versus platinum (Au/Pt) thermocouple, individually calibrated on the International Temperature Scale of 1990 (ITS-90) [2]. This SRM has an overall length of 1.9 m, as measured from the tip of the silica-glass sheath (7.0 mm outer diameter, 71 cm long) to the tip of the stainless steel sheath of the reference-junction probe (6.3 mm outer diameter, 28 cm long). The copper leads from the reference-junction probe of the SRM are 2.1 m long. In use, the reference-junction probe of the SRM is inserted into a properly prepared ice point bath and the copper leads are connected to a high-accuracy voltmeter. The relation of voltage readings of the SRM to the temperature of the measuring junction is provided in this certificate in tabular and mathematical forms.

Certified Values and Confidence Limits: The electromotive force (emf) of the SRM was measured in fixed-point cells at 961.78 °C (silver point), 660.323 °C (aluminum point), 419.527 °C (zinc point), 231.928 °C (tin point), and 156.5985 °C (indium point), and in an ice bath at 0.000 °C, with the reference junctions at the ice point. The measured values of emf are given in Table 1. The emf values computed from the reference function [3] were subtracted from the measured values, and the resulting emf deviations were modeled by a quadratic function of temperature up to 1000 °C. Coefficients of the quadratic function were determined by the method of least squares, and addition of these coefficients to those of the reference function gave the coefficients of the calibration function for this SRM. Table 2 gives corresponding values of the emf of the SRM in millivolts and the temperature of its measuring junction in degrees Celsius when the reference junctions are at 0 °C. Table 3 gives the coefficients of the equation that was used to compute the emf values given in Table 2. Figure 1 shows the deviations of the measured values of emf from values obtained from the Au/Pt reference function. The thermoelectric inhomogeneity of the thermocouple was assessed by measuring its immersion characteristics in the silver and aluminum fixed-point cells [1].

The uncertainty of an emf value can be expressed as an expanded uncertainty, $U = ku_c$, with U determined from a combined standard uncertainty, u_c and a coverage factor, $k = 2$. The expanded uncertainties of the emf values given in Tables 1 and 2 are estimated not to exceed the equivalent of 7 m°C in the range 0 °C to 660 °C, the equivalent of 8 m°C in the range 660 °C to 962 °C, and the equivalent of 14 m°C in the range 962 °C to 1000 °C. These uncertainties include a contribution for the inhomogeneity of the thermocouple.

Construction and calibration of the Au/Pt thermocouple were performed by G.W. Burns and D.C. Ripple of the NIST Process Measurements Division.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by J.W.L. Thomas and J.C. Colbert.

Gaithersburg, MD 20899
Certificate Issue Date: 18 March 1998

Thomas E. Gills, Chief
Standard Reference Materials Program

Expiration of Certification: The certification of this SRM is valid within the measurement uncertainties specified, for at least 1000 h of use at temperatures up to 963 °C, provided the SRM is used in accordance with the Notice and Warning to Users section of this certificate. No long term drift has been observed with several Au/Pt thermocouples that have been studied for periods of time exceeding 1000 h and that have been maintained in accordance with the procedures described in the Instructions for Use. Recertification may be arranged by contacting D.C. Ripple of the NIST Process Measurements Division by phone (301) 975-4801, fax (301) 548-0206, or e-mail dean.ripple@nist.gov. Certification is invalid if the SRM is damaged, contaminated, or modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

Source of Material: The gold wire (Bar 100-431) and the platinum wire (Bar 240-183) for the SRM have a reported purity as a mass fraction of 99.999+ % and were obtained from the Sigmund Cohn Corporation, Mount Vernon, NY. The alumina insulator tubes have a reported purity of 99.8+ % and were obtained from Vesuvius McDanel, Beaver Falls, PA.

NOTICE AND WARNING TO USERS

Storage: SRM 1749 is a thermocouple thermometer, packaged in foam in a wooden case. When not in use, the thermocouple should be stored in its original case to avoid damage.

Handling: This SRM is supplied with a protective silica-glass sheath. If this sheath is damaged, the thermocouple may be subject to contamination with resulting degradations in performance. The thermocouple may be used with or without the protective sheath. However, use of the thermocouple without the protective sheath should be attempted only by trained personnel. Extreme care must be taken to ensure that the expansion coil is not damaged and that the thermocouple is used only in oxidizing environments free of vapors from metals or metal oxides.

Instructions for Use: To use, the copper leads of the SRM should be connected to a high-accuracy voltmeter. To achieve the highest level of accuracy with this SRM, the user should use a calibrated voltmeter with a resolution of 0.01 μV or better, and care must be taken to measure and correct for stray thermal emfs in the measurement circuit.

The reference-junction probe is designed to be inserted through the lid of a standard Dewar flask filled with crushed ice, with distilled water filling the voids between the ice particles. The tip of the reference-junction probe should be immersed 20 cm into the ice-water mixture. The ice should be made from distilled water, the flask should be at least 28 cm deep, and the ice should be lightly packed to the full depth of the flask. After inserting the probe into the ice bath, allow 10 min to 15 min for the probe to attain thermal equilibrium. The reference-junction probe should be kept clean of contaminants that could cause a depression of the melting point of the ice. Washing the probe in distilled water or wiping the probe with a tissue saturated in distilled water will aid in the removal of salts and other impurities.

The silica-glass sheath will devitrify and eventually crack if it is used at temperatures above 500 °C after being contaminated with oils or salts from handling of the sheath with bare hands or other sources. This problem may be avoided by wiping the sheath with a tissue saturated in ethanol or methanol prior to use at elevated temperatures.

The thermocouple should not be used at temperatures exceeding 1000 °C. Higher temperatures may cause slippage of grain boundaries in the gold wire. During use or annealing, not more than 65 cm of the portion of the SRM sheathed in silica glass, as measured from the tip of the sheath, should be heated.

Au/Pt thermocouples are fairly rugged devices, capable of withstanding repeated thermal cycling and small mechanical shocks. However, two maintenance procedures are necessary to ensure that there is no degradation in accuracy.

First, the platinum expansion coil at the measuring junction should be inspected periodically. The silica-glass sheath does not have a matte finish in the vicinity of the measuring junction. This allows the user to inspect the coil without removal of the protective sheath. At room temperature, both thermoelements should extend from the alumina tube by the same amount as when the SRM was first received from NIST. If the protrusion of one of the thermoelements increases by more than about 1 mm after a single period of heating to elevated temperatures and then cooling to room temperature, the thermoelement may have become slightly lodged in the bore of the alumina tube and may have been mechanically strained.

Second, Au/Pt thermocouples should be given a periodic maintenance anneal if they are removed quickly from high temperature environments or if they are used in different thermal environments. The procedure is to anneal the portion of the SRM sheathed in silica glass in a tube furnace at the fullest possible immersion (not more than 65 cm) into the furnace at 1000 °C for 30 min, cool slowly in the furnace to 450 °C, and maintain at 450 °C overnight. The anneal at 1000 °C is optional and only necessary to alleviate strains introduced if the thermocouple has been rapidly cooled.

PREPARATION AND CERTIFICATION MEASUREMENTS

Method of Preparation: After being annealed in air, the Au and Pt thermoelements were inserted into a high purity alumina tube. The measuring junction of the Au/Pt thermocouple was formed by joining the thermoelements at one end with a small coil formed from Pt wire 0.12 mm in diameter. The thermocouple and alumina insulator were mounted inside a protective silica-glass sheath, with the alumina insulator secured in the sheath with a compression fitting. To minimize heat loss by piping of thermal radiation, the silica-glass sheath was given a matte finish. The thermoelements extending from the insulator were covered with coated fiberglass sleeving, and a crimp-type clamp was installed that compresses the sleeving against the thermoelements to anchor them near the end of the alumina insulator. At the end of the thermoelements opposite from the measuring junction, matched copper leads were attached to form the reference junctions. The reference junctions are contained within a stainless steel sheath. To remove residual stresses introduced in the assembly process, the portion of the SRM sheathed in silica glass was given a final furnace anneal.

Measurement Techniques: The emf measurements were made using a calibrated digital multimeter connected to a scanner. Stray thermal emfs from the scanner and voltmeter were carefully measured, and the data were appropriately corrected. Data acquisition was computer controlled. The freezing-point cells used for the calibration of this SRM were all constructed at NIST and intercompared with the reference cells maintained in the NIST Platinum Resistance Thermometer Calibration Laboratory.

COMPATABILITY WITH NIST STANDARD REFERENCE DATABASE 60

The equation in Table 3 is compatible with NIST Standard Reference Database 60, NIST ITS-90 Thermocouple Database. For further information, contact D.C. Ripple of the NIST Process Measurements Division by phone (301) 975-4801, fax (301) 548-0206, or e-mail dean.ripple@nist.gov.

REFERENCES

[1] Burns, G.W. and Ripple, D., "Standard Reference Material 1749: Gold vs. Platinum Thermocouple," NIST Special Publication 260-134, U.S. Government Printing Office, Washington, DC, (1998).

[2] Preston-Thomas, H., "The International Temperature Scale of 1990," Metrologia, 27, pp. 3-10, (1990) and Metrologia, 27, p. 107, (1990).

[3] Burns, G.W., Strouse, G.F., Liu, B.M., and Mangum, B.W., "Gold versus Platinum Thermocouples: Performance Data and an ITS-90 Based Reference Function," in *Temperature: Its Measurement and Control in Science and Industry,* American Institute of Physics, New York, NY, p. 531, (1992).

It is the responsibility of users of this SRM to assure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: Phone (301) 975-6776 (select "Certificates"), Fax (301) 926-4751, e-mail srminfo@nist.gov, or via the Internet http://ts.nist.gov/srm.

TABLE 1. Values of EMF Determined at Fixed Points for the Au/Pt Thermocouple with the Reference Junctions at 0 °C

Temperature (°C, ITS-90)		EMF (mV)
961.78	(silver point)	16.12048
660.323	(aluminum point)	9.32029
419.527	(zinc point)	4.94546
231.928	(tin point)	2.23605
156.5985	(indium point)	1.35079
0.000	(ice point)	–0.00008

TABLE 2. Values of EMF in Millivolts versus Temperature in Degrees Celsius (ITS-90) for the Au/Pt Thermocouple with Reference Junctions at 0 °C

°C	0	1	2	3	4	5	6	7	8	9
	EMF (mV)									
0	−0.0001	0.0060	0.0121	0.0182	0.0244	0.0306	0.0368	0.0431	0.0494	0.0558
10	0.0622	0.0686	0.0751	0.0816	0.0882	0.0947	0.1014	0.1080	0.1147	0.1214
20	0.1282	0.1350	0.1418	0.1487	0.1556	0.1626	0.1696	0.1766	0.1836	0.1907
30	0.1978	0.2050	0.2122	0.2194	0.2267	0.2340	0.2413	0.2487	0.2561	0.2635
40	0.2710	0.2785	0.2860	0.2936	0.3012	0.3088	0.3165	0.3242	0.3320	0.3397
50	0.3475	0.3554	0.3632	0.3711	0.3791	0.3870	0.3950	0.4031	0.4111	0.4192
60	0.4274	0.4355	0.4437	0.4519	0.4602	0.4685	0.4768	0.4852	0.4935	0.5020
70	0.5104	0.5189	0.5274	0.5359	0.5445	0.5531	0.5617	0.5704	0.5791	0.5878
80	0.5965	0.6053	0.6141	0.6230	0.6318	0.6407	0.6497	0.6586	0.6676	0.6766
90	0.6857	0.6948	0.7039	0.7130	0.7222	0.7314	0.7406	0.7498	0.7591	0.7684
100	0.7778	0.7871	0.7965	0.8060	0.8154	0.8249	0.8344	0.8439	0.8535	0.8631
110	0.8727	0.8824	0.8921	0.9018	0.9115	0.9212	0.9310	0.9409	0.9507	0.9606
120	0.9705	0.9804	0.9903	1.0003	1.0103	1.0203	1.0304	1.0405	1.0506	1.0607
130	1.0709	1.0811	1.0913	1.1016	1.1118	1.1221	1.1324	1.1428	1.1532	1.1636
140	1.1740	1.1845	1.1949	1.2054	1.2160	1.2265	1.2371	1.2477	1.2583	1.2690
150	1.2797	1.2904	1.3011	1.3119	1.3227	1.3335	1.3443	1.3552	1.3661	1.3770
160	1.3879	1.3989	1.4098	1.4208	1.4319	1.4429	1.4540	1.4651	1.4762	1.4874
170	1.4986	1.5098	1.5210	1.5323	1.5435	1.5548	1.5661	1.5775	1.5889	1.6003
180	1.6117	1.6231	1.6346	1.6461	1.6576	1.6691	1.6807	1.6923	1.7039	1.7155
190	1.7271	1.7388	1.7505	1.7622	1.7740	1.7857	1.7975	1.8094	1.8212	1.8330
200	1.8449	1.8568	1.8688	1.8807	1.8927	1.9047	1.9167	1.9287	1.9408	1.9529
210	1.9650	1.9771	1.9893	2.0015	2.0136	2.0259	2.0381	2.0504	2.0627	2.0750
220	2.0873	2.0996	2.1120	2.1244	2.1368	2.1493	2.1617	2.1742	2.1867	2.1992
230	2.2118	2.2243	2.2369	2.2495	2.2622	2.2748	2.2875	2.3002	2.3129	2.3257
240	2.3384	2.3512	2.3640	2.3768	2.3897	2.4025	2.4154	2.4283	2.4413	2.4542
250	2.4672	2.4802	2.4932	2.5062	2.5193	2.5323	2.5454	2.5586	2.5717	2.5848
260	2.5980	2.6112	2.6244	2.6377	2.6509	2.6642	2.6775	2.6908	2.7042	2.7175
270	2.7309	2.7443	2.7577	2.7712	2.7846	2.7981	2.8116	2.8251	2.8387	2.8523
280	2.8658	2.8794	2.8931	2.9067	2.9204	2.9340	2.9477	2.9615	2.9752	2.9890
290	3.0027	3.0165	3.0304	3.0442	3.0581	3.0719	3.0858	3.0997	3.1137	3.1276
300	3.1416	3.1556	3.1696	3.1836	3.1977	3.2118	3.2259	3.2400	3.2541	3.2682
310	3.2824	3.2966	3.3108	3.3250	3.3393	3.3535	3.3678	3.3821	3.3964	3.4108
320	3.4251	3.4395	3.4539	3.4683	3.4827	3.4972	3.5117	3.5262	3.5407	3.5552
330	3.5697	3.5843	3.5989	3.6135	3.6281	3.6427	3.6574	3.6721	3.6868	3.7015
340	3.7162	3.7310	3.7457	3.7605	3.7753	3.7901	3.8050	3.8198	3.8347	3.8496
350	3.8645	3.8795	3.8944	3.9094	3.9244	3.9394	3.9544	3.9695	3.9845	3.9996
360	4.0147	4.0298	4.0449	4.0601	4.0753	4.0904	4.1057	4.1209	4.1361	4.1514
370	4.1667	4.1820	4.1973	4.2126	4.2279	4.2433	4.2587	4.2741	4.2895	4.3050
380	4.3204	4.3359	4.3514	4.3669	4.3824	4.3980	4.4135	4.4291	4.4447	4.4603
390	4.4760	4.4916	4.5073	4.5230	4.5387	4.5544	4.5701	4.5859	4.6017	4.6174
400	4.6333	4.6491	4.6649	4.6808	4.6967	4.7126	4.7285	4.7444	4.7604	4.7763

TABLE 2 (Continued). Values of EMF in Millivolts versus Temperature in Degrees Celsius (ITS-90) for the Au/Pt Thermocouple with Reference Junctions at 0 °C

°C	0	1	2	3	4	5	6	7	8	9
	EMF (mV)									
400	4.6333	4.6491	4.6649	4.6808	4.6967	4.7126	4.7285	4.7444	4.7604	4.7763
410	4.7923	4.8083	4.8243	4.8404	4.8564	4.8725	4.8886	4.9047	4.9208	4.9369
420	4.9531	4.9693	4.9855	5.0017	5.0179	5.0341	5.0504	5.0667	5.0830	5.0993
430	5.1156	5.1320	5.1483	5.1647	5.1811	5.1975	5.2140	5.2304	5.2469	5.2634
440	5.2799	5.2964	5.3129	5.3295	5.3460	5.3626	5.3792	5.3958	5.4125	5.4291
450	5.4458	5.4625	5.4792	5.4959	5.5126	5.5294	5.5462	5.5630	5.5798	5.5966
460	5.6134	5.6303	5.6472	5.6640	5.6810	5.6979	5.7148	5.7318	5.7488	5.7657
470	5.7828	5.7998	5.8168	5.8339	5.8510	5.8680	5.8852	5.9023	5.9194	5.9366
480	5.9538	5.9710	5.9882	6.0054	6.0226	6.0399	6.0572	6.0745	6.0918	6.1091
490	6.1264	6.1438	6.1612	6.1786	6.1960	6.2134	6.2308	6.2483	6.2658	6.2833
500	6.3008	6.3183	6.3359	6.3534	6.3710	6.3886	6.4062	6.4238	6.4415	6.4591
510	6.4768	6.4945	6.5122	6.5299	6.5477	6.5654	6.5832	6.6010	6.6188	6.6366
520	6.6545	6.6723	6.6902	6.7081	6.7260	6.7439	6.7618	6.7798	6.7978	6.8158
530	6.8338	6.8518	6.8698	6.8879	6.9060	6.9241	6.9422	6.9603	6.9784	6.9966
540	7.0147	7.0329	7.0511	7.0693	7.0876	7.1058	7.1241	7.1424	7.1607	7.1790
550	7.1973	7.2157	7.2341	7.2525	7.2709	7.2893	7.3077	7.3261	7.3446	7.3631
560	7.3816	7.4001	7.4186	7.4372	7.4558	7.4743	7.4929	7.5115	7.5302	7.5488
570	7.5675	7.5862	7.6049	7.6236	7.6423	7.6610	7.6798	7.6986	7.7174	7.7362
580	7.7550	7.7738	7.7927	7.8116	7.8305	7.8494	7.8683	7.8872	7.9062	7.9252
590	7.9442	7.9632	7.9822	8.0012	8.0203	8.0393	8.0584	8.0775	8.0967	8.1158
600	8.1349	8.1541	8.1733	8.1925	8.2117	8.2309	8.2502	8.2695	8.2887	8.3080
610	8.3274	8.3467	8.3660	8.3854	8.4048	8.4242	8.4436	8.4630	8.4825	8.5019
620	8.5214	8.5409	8.5604	8.5799	8.5995	8.6190	8.6386	8.6582	8.6778	8.6974
630	8.7171	8.7367	8.7564	8.7761	8.7958	8.8155	8.8352	8.8550	8.8748	8.8945
640	8.9144	8.9342	8.9540	8.9739	8.9937	9.0136	9.0335	9.0534	9.0734	9.0933
650	9.1133	9.1332	9.1532	9.1733	9.1933	9.2133	9.2334	9.2535	9.2736	9.2937
660	9.3138	9.3339	9.3541	9.3743	9.3945	9.4147	9.4349	9.4551	9.4754	9.4957
670	9.5159	9.5363	9.5566	9.5769	9.5973	9.6176	9.6380	9.6584	9.6788	9.6993
680	9.7197	9.7402	9.7607	9.7812	9.8017	9.8222	9.8428	9.8633	9.8839	9.9045
690	9.9251	9.9457	9.9664	9.9870	10.0077	10.0284	10.0491	10.0698	10.0906	10.1113
700	10.1321	10.1529	10.1737	10.1945	10.2154	10.2362	10.2571	10.2780	10.2989	10.3198
710	10.3407	10.3617	10.3827	10.4036	10.4246	10.4457	10.4667	10.4877	10.5088	10.5299
720	10.5510	10.5721	10.5932	10.6144	10.6355	10.6567	10.6779	10.6991	10.7203	10.7416
730	10.7628	10.7841	10.8054	10.8267	10.8480	10.8694	10.8907	10.9121	10.9335	10.9549
740	10.9763	10.9977	11.0192	11.0406	11.0621	11.0836	11.1051	11.1267	11.1482	11.1698
750	11.1914	11.2130	11.2346	11.2562	11.2778	11.2995	11.3212	11.3429	11.3646	11.3863
760	11.4080	11.4298	11.4516	11.4734	11.4952	11.5170	11.5388	11.5607	11.5825	11.6044
770	11.6263	11.6482	11.6702	11.6921	11.7141	11.7361	11.7581	11.7801	11.8021	11.8241
780	11.8462	11.8683	11.8904	11.9125	11.9346	11.9568	11.9789	12.0011	12.0233	12.0455
790	12.0677	12.0899	12.1122	12.1345	12.1567	12.1790	12.2014	12.2237	12.2460	12.2684
800	12.2908	12.3132	12.3356	12.3580	12.3805	12.4029	12.4254	12.4479	12.4704	12.4929

TABLE 2 (Continued). Values of EMF in Millivolts versus Temperature in Degrees Celsius (ITS-90) for the Au/Pt Thermocouple with Reference Junctions at 0 °C

°C	0	1	2	3	4	5	6	7	8	9
					EMF (mV)					
800	12.2908	12.3132	12.3356	12.3580	12.3805	12.4029	12.4254	12.4479	12.4704	12.4929
810	12.5155	12.5380	12.5606	12.5832	12.6058	12.6284	12.6511	12.6737	12.6964	12.7191
820	12.7418	12.7645	12.7872	12.8100	12.8327	12.8555	12.8783	12.9011	12.9240	12.9468
830	12.9697	12.9925	13.0154	13.0383	13.0613	13.0842	13.1072	13.1301	13.1531	13.1761
840	13.1991	13.2222	13.2452	13.2683	13.2914	13.3145	13.3376	13.3607	13.3839	13.4070
850	13.4302	13.4534	13.4766	13.4998	13.5231	13.5463	13.5696	13.5929	13.6162	13.6395
860	13.6628	13.6862	13.7096	13.7329	13.7563	13.7798	13.8032	13.8266	13.8501	13.8736
870	13.8971	13.9206	13.9441	13.9677	13.9912	14.0148	14.0384	14.0620	14.0856	14.1092
880	14.1329	14.1566	14.1802	14.2039	14.2277	14.2514	14.2751	14.2989	14.3227	14.3465
890	14.3703	14.3941	14.4180	14.4418	14.4657	14.4896	14.5135	14.5374	14.5613	14.5853
900	14.6093	14.6332	14.6572	14.6813	14.7053	14.7293	14.7534	14.7775	14.8016	14.8257
910	14.8498	14.8739	14.8981	14.9223	14.9465	14.9707	14.9949	15.0191	15.0434	15.0676
920	15.0919	15.1162	15.1405	15.1649	15.1892	15.2136	15.2380	15.2623	15.2868	15.3112
930	15.3356	15.3601	15.3845	15.4090	15.4335	15.4581	15.4826	15.5071	15.5317	15.5563
940	15.5809	15.6055	15.6301	15.6548	15.6794	15.7041	15.7288	15.7535	15.7782	15.8030
950	15.8277	15.8525	15.8773	15.9021	15.9269	15.9517	15.9766	16.0014	16.0263	16.0512
960	16.0761	16.1010	16.1260	16.1509	16.1759	16.2009	16.2259	16.2509	16.2759	16.3010
970	16.3261	16.3511	16.3762	16.4013	16.4265	16.4516	16.4768	16.5020	16.5271	16.5524
980	16.5776	16.6028	16.6281	16.6533	16.6786	16.7039	16.7292	16.7546	16.7799	16.8053
990	16.8307	16.8561	16.8815	16.9069	16.9323	16.9578	16.9833	17.0088	17.0343	17.0598
1000	17.0853									

TABLE 3. Coefficients of the Equation Used to Compute the EMF Values Given in Table 2

The equation for computing values of emf as a function of temperature is of the form:

$$E = a_0 + a_1(t_{90}) + a_2(t_{90})^2 + a_3(t_{90})^3 + a_4(t_{90})^4 + a_5(t_{90})^5 + a_6(t_{90})^6 + a_7(t_{90})^7 + a_8(t_{90})^8 + a_9(t_{90})^9,$$

where E is the emf in millivolts and t_{90} is the temperature in degrees Celsius (ITS-90). The coefficients for the indicated temperature range are:

		0 °C to 1000 °C
a_0	=	–0.829775530E–04
a_1	=	0.603577729E–02
a_2	=	0.193678032E–04
a_3	=	–0.222998614E–07
a_4	=	0.328711859E–10
a_5	=	–0.424206193E–13
a_6	=	0.456927038E–16
a_7	=	–0.339430259E–19
a_8	=	0.142981590E–22
a_9	=	–0.251672787E–26

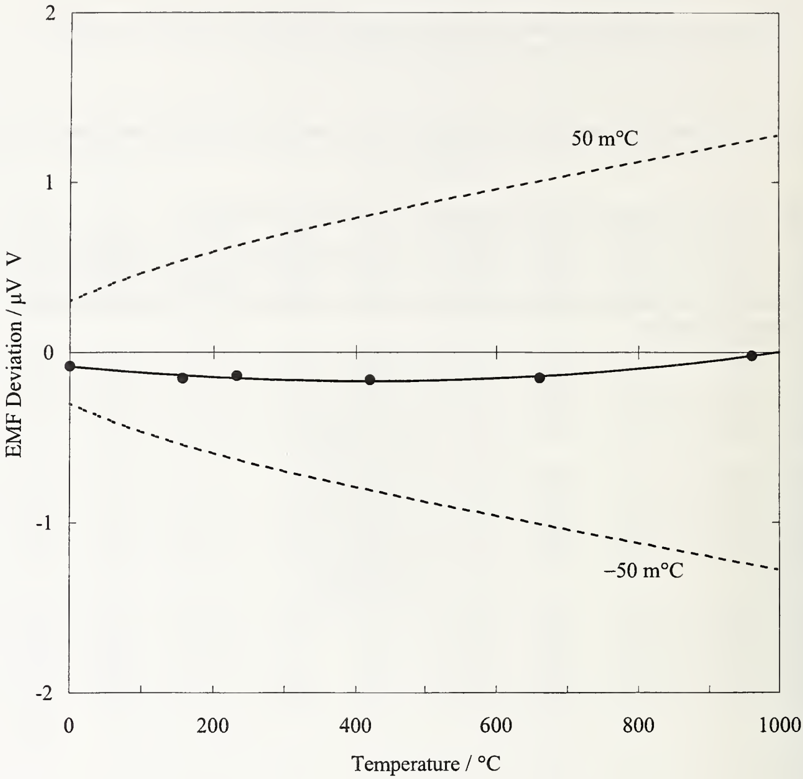

Figure 1: This figure shows the deviation of the emf of the Au/Pt thermocouple from an emf/temperature reference function for Au/Pt thermocouples given in Reference [3]. The deviation equals the thermocouple emf minus the reference function emf. The dots denote measured values and the solid curve is calculated from a polynomial fitted to the data by the method of least squares. The dashed lines indicate an emf deviation equivalent to 0.05 °C

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